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November 8, 1990

Ms. Debbie Rossi
U.S. Environmental Protection Agency
Region III
841 Chestnut Building
Philadelphia, PA 19107

Re: Quality Assurance Project Plan Addendum/
Update for Phase III Analytical Program
Woodlawn Landfill RI/FS, Cecil County, Maryland

Dear Ms. Rossi:

This letter addendum updates the existing Quality Assurance Project Plan (QAPP) (Revision 05, November 30, 1989) to include the revised analytical program for groundwater at the Woodlawn Landfill in Cecil County, Maryland. As approved by the Environmental Protection Agency (U.S. EPA) Region III on October 18, 1990 (letter from Debbie Rossi), the analytical program was modified to include the analysis of vinyl chloride using U.S. EPA Method 502.2 ("Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Photoionization and Electrolytic Conductivity Detectors in Series;" EPA-600/4-88/039, September 1986).

This method will be used in addition to the existing Contract Laboratory Program (CLP) protocol. It was chosen to provide a lower detection limit (0.18 ug/l) for vinyl chloride. EPA Method 502.2, provided in the attached document, is summarized below as follows:

"Highly volatile organic compounds with low water solubility are extracted (purged) from the sample matrix by bubbling an inert gas through a 5 ml aqueous sample. Purged sample components are trapped in a tube containing suitable sorbent materials. When purging is complete, the sorbent tube is heated and backflushed with helium to desorb trapped sample components onto a capillary gas chromatography (GC) column. The column is temperature programmed to separate the method analytes which are then detected with a photoionization detector (PID) and a halogen specific detector placed in series. Tentative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing resultant GC retention times. Additional confirmatory information can be gained by comparing the relative response from the two detectors. Each identified component is measured by

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relating the response produced for that compound to the response produced by a compound that is used as an internal standard."

International Technology Analytical Services (ITAS) in San Jose, California (2055 Junction Avenue, San Jose, CA 95131) has been selected to perform the analysis of groundwater using U.S. EPA Method 502.2. This laboratory meets the same Quality Assurance/Quality Control (QA/QC) objectives as the approved ITAS-Export, Pennsylvania laboratory, as per the Quality Assurance Manual Laboratory-Specific Attachment (Document: ITAS/SJ/QAM, Revision 2.0, September 29, 1989).

In keeping with the basic format of the existing QAPP, the following QC data from the ITAS-San Jose, California laboratory is provided for Method 502.2:

- Precision (Relative Percent Difference [RPD]) for vinyl chloride - 11.8
- Accuracy (percent Recovery) for vinyl chloride - 111.0

All aspects of groundwater sample collection procedures, sample custody, equipment calibration, analytical procedures, and QA/QC will be performed in accordance with the existing QAPP and/or the methods documentation for the determination of organic compounds in drinking water (U.S. EPA Method 502.2).

Sincerely yours,

George B. Markert/AMJ

George B. Markert
Corporate Environmental Affairs

GBM:AMJ:jar
Enclosures

cc: Mr. Milton Marder, Maryland Department of Environment
Mr. Barry Belford, Cecil County, Department of Public Works
Mr. Mark Grummer, Kirkland and Ellis

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METHOD 502.2. VOLATILE ORGANIC COMPOUNDS IN WATER BY
PURGE AND TRAP CAPILLARY COLUMN GAS CHROMATOGRAPHY
WITH PHOTOIONIZATION AND ELECTROLYTIC
CONDUCTIVITY DETECTORS IN SERIES

(September, 1986)

1. SCOPE AND APPLICATION

- 1.1 This method is applicable for the determination of various volatile organic compounds in finished drinking water, raw source water, or drinking water in any treatment stage. (1,2) The following compounds can be determined by this method:

<u>Analyte</u>	<u>Chemical Abstract Services Registry Number</u>
/Benzene	71-43-2
/Bromobenzene	108-86-1
/Bromochloromethane	74-97-5
/Bromodichloromethane	75-27-4
/Bromoform	75-25-2
/Bromomethane	74-83-9
/n-Butylbenzene	104-51-8
/sec-Butylbenzene	135-98-8
/tert-Butylbenzene	98-06-6
/Carbon tetrachloride	56-23-5
/Chlorobenzene	108-90-7
/Chloroethane	75-00-3
/Chloroform	67-66-3
/Chloromethane	74-87-3
/2-Chlorotoluene	95-49-8
/4-Chlorotoluene	106-43-4
/Dibromochloromethane	124-48-1
/1,2-Dibromo-3-chloropropane	96-12-8
/1,2-Dibromoethane	106-93-4
/Dibromomethane	74-95-3
/1,2-Dichlorobenzene	95-50-1
/1,3-Dichlorobenzene	541-73-1
/1,4-Dichlorobenzene	106-46-7
/Dichlorodifluoromethane	75-71-8
/1,1-Dichloroethane	75-34-3
/1,2-Dichloroethane	107-06-2
/1,1-Dichloroethene	75-35-4
/cis-1,2-Dichloroethene	156-59-4

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Analyte	Chemical Abstract Services Registry Number
-trans-1,2-Dichloroethene	156-60-5
/1,2-Dichloropropane	78-87-5
-1,3-Dichloropropane	142-28-9
-2,2-Dichloropropane	590-20-7
-1,1-Dichloropropene	563-58-6
/Ethylbenzene	100-41-4
/Hexachlorobutadiene	87-68-3
/Isopropylbenzene (i-c-n-c)	98-82-8
(u) /p-Isopropyltoluene (p-c-n-c)	99-87-6
/Methylene chloride	75-09-2
/Naphthalene	91-20-3
/n-Propylbenzene	103-65-1
/Styrene	100-42-5
-1,1,1,2-Tetrachloroethane	630-20-6
/1,1,2,2-Tetrachloroethane	79-34-5
/Tetrachloroethene	127-18-4
/Toluene	108-88-3
/1,2,3-Trichlorobenzene	87-61-6
/1,2,4-Trichlorobenzene	120-82-1
/1,1,1-Trichloroethane	71-55-6
/1,1,2-Trichloroethane	79-00-5
/Trichloroethene	79-01-6
/Trichlorofluoromethane	75-69-4
/1,2,3-Trichloropropane	96-18-4
/1,2,4-Trimethylbenzene	95-63-6
/1,3,5-Trimethylbenzene	108-67-8
/Vinyl chloride	75-01-4
-o-Xylene	95-47-6
/m-Xylene	108-38-3
/p-Xylene	106-42-3

- 1.2 Method detection limits (MDLs) (3) are compound dependent and vary with purging efficiency and concentration. The MDLs for selected analytes are presented in Table 1. The applicable concentration range of this method is compound and instrument dependent but is approximately 0.1 to 200 µg/L. Analytes that are inefficiently purged from water will not be detected when present at low concentrations, but they can be measured with acceptable accuracy and precision when present in sufficient amounts. Determination of some structural isomers (i.e., xylenes) may be hampered by coelution.

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- 1.3 This method is recommended for use only by analysts experienced in the measurement of purgeable organics at the low $\mu\text{g/L}$ level or by experienced technicians under the close supervision of a qualified analyst.

2. SUMMARY OF METHOD

- 2.1 Highly volatile organic compounds with low water solubility are extracted (purged) from the sample matrix by bubbling an inert gas through a 5 mL aqueous sample. Purged sample components are trapped in a tube containing suitable sorbent materials. When purging is complete, the sorbent tube is heated and backflushed with helium to desorb trapped sample components onto a capillary gas chromatography (GC) column. The column is temperature programmed to separate the method analytes which are then detected with a photoionization detector (PID) and a halogen specific detector placed in series.
- 2.2 Tentative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing resultant GC retention times. Additional confirmatory information can be gained by comparing the relative response from the two detectors. Each identified component is measured by relating the response produced for that compound to the response produced by a compound that is used as an internal standard.

3. INTERFERENCES

- 3.1 During analysis, major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) plastic tubing, non-PTFE thread sealants, or flow controllers with rubber components in the purging device should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of laboratory reagent blanks (Sect. 9.1.3) provide information about the presence of contaminants. When potential interfering peaks are noted in laboratory reagent blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter (Fig. 1). Subtracting blank values from sample results is not permitted.
- 3.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing relatively high concentrations of volatile organic compounds. A preventive technique is between-sample rinsing of the purging apparatus and sample syringes with two portions of reagent water. After analysis of a sample containing high concentrations of volatile organic compounds, one or more laboratory reagent blanks should be analyzed to check for cross contamination. For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds or high levels of compounds being determined, it may be

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necessary to wash out the purging device with a soap solution, rinse it with reagent water, and then dry it in an oven at 105°C between analyses.

- 3.3 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean since clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination.

4. SAFETY

- 4.1 The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Each laboratory is responsible for maintaining awareness of OSHA regulations regarding safe handling of chemicals used in this method. Additional references to laboratory safety are available (4-6) for the information of the analyst.
- 4.2 The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon-tetrachloride, 1,4-dichlorobenzene, 1,2-dichlorethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. APPARATUS AND EQUIPMENT

- 5.1 SAMPLE CONTAINERS - 40-mL to 120-mL screw cap vials (Pierce #13075 or equivalent) each equipped with a PTFE-faced silicone septum (Pierce #12722 or equivalent). Prior to use, wash vials and septa with detergent and rinse with tap and distilled water. Allow the vials and septa to air dry at room temperature, place in a 105°C oven for one hour, then remove and allow to cool in an area known to be free of organics.
- 5.2 PURGE AND TRAP SYSTEM - The purge and trap system consists of three separate pieces of equipment: purging device, trap, and desorber. Systems are commercially available from several sources that meet all of the following specifications.

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- 5.2.1 The all glass purging device (Fig. 1) must be designed to accept 5-mL samples with a water column at least 5 cm deep. Gaseous volumes above the sample must be kept to a minimum (< 15 mL) to eliminate dead volume effects. A glass frit should be installed at the base of the sample chamber so that the purge gas passes through the water column as finely divided bubbles with a diameter of < 3 mm at the origin. Needle spargers may be used, however, the purge gas must be introduced at a point \leq 5 mm from the base of the water column.
- 5.2.2 The trap (Fig. 2) must be at least 25 cm long and have an inside diameter of at least 0.105 in. Starting from the inlet, the trap should contain 1.0 cm of methyl silicone coated packing and the following amounts of adsorbents: 1/3 of 2,6-diphenylene oxide polymer, 1/3 of silica gel, and 1/3 of coconut charcoal. Before initial use, the trap should be conditioned overnight at 180°C by backflushing with an inert gas flow of at least 6 mL/min. Vent the trap effluent to the room, not to the analytical column. Prior to use, the trap should be conditioned for 10 minutes at 180°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.
- 5.2.3 The use of the methyl silicone coated packing is recommended, but not mandatory. The packing serves a dual purpose of protecting the adsorbent from aerosols, and also of insuring that the adsorbent is fully enclosed within the heated zone of the trap thus eliminating potential cold spots. Alternatively, silanized glass wool may be used as a spacer at the trap inlet.
- 5.2.4 The desorber must be capable of rapidly heating the trap to 180°C. The polymer section of the trap should not be heated higher than 200°C or the life expectancy of the trap will decrease. Trap failure is characterized by a pressure drop in excess of 3 pounds per square inch across the trap during purging or by poor bromoform sensitivities. The desorber design illustrated in Fig. 2 meets these criteria.
- 5.2.5 Figures 3 and 4 show typical flow patterns for the purge-sorb and desorb modes.

5.3 GAS CHROMATOGRAPH

- 5.3.1 The GC must be capable of temperature programming and should be equipped with variable-constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program

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operation. The column oven must be cooled to $<10^{\circ}\text{C}$ (Sect. 5.3.2), therefore, a subambient oven controller is required. The carrier gas flow is augmented with an additional 24 mL of helium flow before entering the photoionization detector. This make-up gas is necessary to ensure optimal response from both detectors. The photoionization and electroconductivity detector are connected with a short piece of uncoated capillary tubing, 0.32 - 0.5mm ID.

5.3.2 Gas Chromatographic Column - 60m long x 0.75mm ID VOCOL (Supelco, Inc.) wide-bore capillary column with 1.5 μm film thickness, or equivalent. The flow rate of helium carrier gas is adjusted to about 6 mL/min. The column temperature is held for 8 minutes at 10°C , then programmed to 180°C at $4^{\circ}\text{C}/\text{min}$, and held until all expected compounds have eluted. A sample chromatogram obtained with this column is presented in Fig. 5. This column was used to develop the method performance statements in Section 12.

5.3.3 A high temperature photoionization detector equipped with a 10.2 eV lamp is required (Tracor Model 703 or equivalent).

5.3.4 An electrolytic conductivity or microcoulometric detector is required. These halogen-specific systems eliminate misidentifications due to non-organohalides which are coextracted during the purge step. A Tracor Hall Model 700-A detector was used to gather the single laboratory accuracy and precision data. The operating conditions used to collect these data are as follow:

Reactor tube:	Nickel 1/16 in OD
Reactor temperature:	810°C
Reactor base temperature:	250°C
Electrolyte:	100% n-propyl alcohol
Electrolyte flow rate:	0.8 mL/min
Reaction gas:	Hydrogen at 40 mL/min
Carrier gas plus make-up gas:	Helium at 30 mL/min

5.4 SYRINGE AND SYRINGE VALVES

5.4.1 Two 5-mL glass hypodermic syringes with Luer-Lok tip.

5.4.2 Three 2-way syringe valves with Luer ends.

5.4.3 One 25- μL micro syringe with a 2 in x 0.006 in ID, 22° bevel needle (Hamilton #702N or equivalent).

5.4.4 Micro syringes - 10, 100 μL .

5.4.5 Syringes - 0.5, 1.0, and 5-mL, gas tight with shut-off valve.

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5.5 MISCELLANEOUS

- 5.5.1 Standard solution storage containers - 15-mL bottles with PTFE-lined screw caps.

6. REAGENTS AND CONSUMABLE MATERIALS

6.1 TRAP PACKING MATERIALS

- 6.1.1 2,6-Diphenylene oxide polymer, 60/80 mesh, chromatographic grade (Tenax GC or equivalent).
- 6.1.2 Methyl silicone packing (optional) - OV-1 (3%) on Chromosorb W, 60/80 mesh, or equivalent.
- 6.1.3 Silica gel - 35/60 mesh, Davison, grade 15 or equivalent.
- 6.1.4 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26 lot #M-2649 by crushing through 26 mesh screen.

6.2 REAGENTS

- 6.2.1 Methanol - Demonstrated to be free of analytes.
- 6.2.2 Reagent water - Prepare reagent water by passing tap water through a filter bed containing about 0.5 kg of activated carbon, by using a water purification system, or by boiling distilled water for 15 min followed by a 1-h purge with inert gas while the water temperature is held at 90°C. Store in clean, narrow-mouth bottles with PTFE-lined septa and screw caps.
- 6.2.3 Hydrochloric acid (1+1) - Carefully add a measured volume of conc. HCl to equal volume of reagent water.
- 6.2.4 Vinyl chloride - 99.9% pure vinyl chloride is available from Ideal Gas Products, Inc., Edison, New Jersey and from Matheson, East Rutherford, New Jersey. Certified mixtures of vinyl chloride in nitrogen at 1.0 and 10.0 ppm (v/v) are available from several sources.

- 6.3 STANDARD STOCK SOLUTIONS - These solutions may be purchased as certified solutions or prepared from pure standard materials using the following procedures:

- 6.3.1 Place about 9.8 mL of methanol into a 10-mL ground-glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol-wetted surfaces have dried and weigh to the nearest 0.1 mg.

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- 6.3.2 If the analyte is a liquid at room temperature, use a 100- μ L syringe and immediately add two or more drops of reference standard to the flask. Be sure that the reference standard falls directly into the alcohol without contacting the neck of the flask. If the analyte is a gas at room temperature, fill a 5-mL valved gas-tight syringe with the standard to the 5.0-mL mark, lower the needle to 5 mm above the methanol meniscus, and slowly inject the standard into the neck area of the flask. The gas will rapidly dissolve in the methanol.
- 6.3.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter from the net gain in weight. When compound purity is certified at 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard.
- 6.3.4 Store stock standard solutions in 15-mL bottles equipped with PTFE-lined screw caps. Methanol solutions prepared from liquid analytes are stable for at least four weeks when stored at 4°C. Methanol solutions prepared from gaseous analytes are not stable for more than one week when stored at <0°C; at room temperature, they must be discarded after one day.
- 6.4 SECONDARY DILUTION STANDARDS - Use standard stock solutions to prepare secondary dilution standard solutions that contain the analytes in methanol. The secondary dilution standards should be prepared at concentrations that can be easily diluted to prepare aqueous calibration solutions (Sect. 8.1) that will bracket the working concentration range. Store the secondary dilution standard solutions with minimal headspace and check frequently for signs of deterioration or evaporation, especially just before preparing calibration solutions from them. Storage times described for stock standard solutions in Sect. 6.3.4 also apply to secondary dilution standard solutions.
- 6.5 INTERNAL STANDARD SPIKING SOLUTION — Prepare a spiking solution containing fluorobenzene, and 2-bromo-1-chloropropane in methanol using the procedures described in Sect. 6.3 and 6.4. It is recommended that the secondary dilution standard be prepared at a concentration of 5 μ g/mL of each internal standard compound. The addition of 10 μ L of such a standard to 5.0 mL of sample or calibration standard would be equivalent to 10 μ g/L.
- 6.6 LABORATORY QUALITY CONTROL CHECK STANDARD CONCENTRATE - Using standard stock solutions, prepare a solution containing each analyte of interest at a concentration of 100 times the maximum contaminant level (MCL) or 1 μ g/mL, whichever is smaller, in methanol.

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7. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

7.1 SAMPLE COLLECTION

- 7.1.1 Collect all samples in duplicate. Fill sample bottles to overflowing. No air bubbles should pass through the sample as the bottle is filled, or be trapped in the sample when the bottle is sealed.
- 7.1.2 When sampling from a water tap, open the tap and allow the system to flush until the water temperature has stabilized (usually about 10 min). Adjust the flow to about 500 mL/min and collect duplicate samples from the flowing stream.
- 7.1.3 When sampling from an open body of water, fill a 1-quart wide-mouth bottle or 1-liter beaker with sample from a representative area, and carefully fill duplicate sample bottles from the container.

7.2 SAMPLE PRESERVATION

- 7.2.1 Adjust the pH of the duplicate samples to <2 by carefully adding one drop of 1:1 HCl for each 20 mL of sample volume.(7) Seal the sample bottles, PTFE-face down, and shake vigorously for one minute.
- 7.2.2 The samples must be chilled to 4°C on the day of collection and maintained at that temperature until analysis. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure that they will be at 4°C on arrival at the laboratory.

7.3 SAMPLE STORAGE

- 7.3.1 Store samples at 4°C until analysis. The sample storage area must be free of organic solvent vapors.
- 7.3.2 Analyze all samples within 14 days of collection. Samples not analyzed within this period must be discarded and replaced.

8. CALIBRATION AND STANDARDIZATION

8.1 PREPARATION OF CALIBRATION STANDARDS

- 8.1.1 A set of at least five calibration standards containing the method analytes is needed. One calibration standard should contain each analyte at a concentration approaching but greater than the method detection limit (Table 1) for that compound; the other calibration standards should contain analytes at concentrations that define the range of the method or the detection system.

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8.1.2 To prepare a calibration standard, add an appropriate volume of a secondary dilution standard solution to an aliquot of reagent water in a volumetric flask. Use a microsyringe and rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the flask three times only. Discard the contents contained in the neck of the flask. Aqueous standards are not stable and should be discarded after one hour unless sealed and stored as described in Sect. 7.2.

8.2 CALIBRATION

8.2.1 Analyze each calibration standard according to Sect. 10, adding 10 μ L of internal standard spiking solution directly to the syringe. Tabulate area response versus the concentration for each analyte and internal standard. Calculate response factors (RF) for each analyte using Equation 1:

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)} \quad \text{Equation 1}$$

where:

A_s = Area for the analyte to be measured;
 A_{is} = Area for the internal standard;
 C_{is} = Concentration of the internal standard, in μ g/L.
 C_s = Concentration of the analyte to be measured, in μ g/L.

The choice of which internal standard to use for an analyte is left to the analyst.

8.2.2 Prepare a calibration curve for each analyte. Alternatively, if the RF for an analyte is constant (less than 15% RSD) over the working range, the average RF can be used for that analyte.

8.2.3 The working calibration curve or average response factor must be verified on each working day by the measurement of one or more calibration standards. If the area for any analyte varies from the response determined for that standard concentration from the calibration curve or average RF established in Sect. 8.2.2 by more than $\pm 20\%$, repeat steps 8.2.1 and 8.2.2.

8.2.4 Calibration for vinyl chloride using a certified gaseous mixture of vinyl chloride in nitrogen can be accomplished by the following steps. The procedure requires use of a modified purge vessel having a septum seal port in the expanded part of the device.

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8.2.4.1 Fill the purging device with 5.0 mL of reagent water or aqueous calibration standard, and add internal standards.

8.2.4.2 Start to purge the aqueous mixture. Inject a known volume (between 100 and 2000 μ L) of the calibration gas (at room temperature) directly into the purging device with a gas tight syringe. Slowly inject the gaseous sample through a septum seal at the top of the purging device at 2000 μ L/min. Do not inject the standard through the aqueous sample inlet needle. Inject the gaseous standard before five min of the 11-min purge time have elapsed.

8.2.4.3 Determine the aqueous equivalent concentration of vinyl chloride standard injected in μ g/L, according to Equation 2:

$$S = 0.510 (C)(V) \quad \text{Equation 2}$$

where S = Aqueous equivalent concentration of vinyl chloride standard in μ g/L;
C = Concentration of gaseous standard in ppm (v/v);
V = Volume of standard injected in milliliters.

9. QUALITY CONTROL

9.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. A quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

9.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 9.2.

9.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Section 10.1.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 9.2.

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- 9.1.3 Each day, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system are under control.
- 9.1.4 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 9.3. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but at least two samples per month.
- 9.1.5 On a weekly basis, the laboratory must demonstrate the ability to analyze low level samples. A procedure for low level check samples is described in Section 9.4.
- 9.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 9.5.
- 9.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.
- 9.2.1 A quality control (QC) check sample concentrate is required containing each regulated analyte, and any additional analyte which is to be reported, at a concentration of 100 times the MCL or 1 $\mu\text{g/mL}$, whichever is smaller, in methanol. The QC check sample must be prepared by the laboratory using stock standards prepared independently from those used for calibration.
- 9.2.2 Analyze seven 5-mL QC check samples at 1/5 MCL or 2 $\mu\text{g/L}$ according to the method beginning in Sect. 10. Each sample is produced by injecting 10 μL of QC check sample concentrate into 5 mL of reagent water in a glass syringe through the syringe valve.
- 9.2.3 Calculate the average recovery (\bar{X}) in $\mu\text{g/L}$, and the standard deviation of the recovery (s) in $\mu\text{g/L}$ for each analyte using the seven results. Calculate the MDL for each analyte as specified in Ref. 2. The calculated MDL must be less than the spike level.
- 9.2.4 For each analyte, (\bar{X}) must be between 80% and 120% of the true value. Additionally, s must be $\leq 30\%$ of \bar{X} . If s and \bar{X} for all analytes meet the criteria, the system performance is acceptable and analysis of actual samples can begin. If any s exceeds the precision limit or any \bar{X} falls outside the range for accuracy, the system performance is unacceptable for that analyte.

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NOTE: The large number of analytes present a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes are determined.

- 9.2.5 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must repeat the test according to Section 9.2.2 only for the analytes which failed the test.
- 9.3 The laboratory must demonstrate on a regular basis, as outlined in Sect. 9.1.4, that the measurement system is in control by analyzing a quality control sample for all analytes of interest at the MCL or 10 $\mu\text{g/L}$, whichever is smaller.
- 9.3.1 Prepare a QC check standard by adding 50 μL of QC check sample concentrate to 5 mL of reagent water in a glass syringe.
- 9.3.2 Analyze the QC check according to Section 10, and calculate the recovery for each analyte. The recovery must be between 60% and 140% of the expected value.
- 9.3.3 If the recovery for any analyte falls outside the designated range, the analyte has failed the acceptance criteria. A check standard containing each failed analyte must be re-analyzed.
- 9.4 On a weekly basis, the laboratory must demonstrate the ability to analyze low level samples.
- 9.4.1 Prepare a low level check sample by adding 10 μL of QC check sample concentrate to 5 mL of reagent water in a 5 mL syringe. Analyze according to the method in Sect. 10.
- 9.4.2 For each analyte, the recovery must be between 60% and 140% of the expected value.
- 9.4.3 When one or more analytes fail the test, the analyst must repeat the test only for those analytes which failed to meet the criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 9.4.1.
- 9.5 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

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10. PROCEDURE

10.1 INITIAL CONDITIONS

10.1.1 Recommended chromatographic conditions are summarized in Section 5.3.2. Estimated retention times and MDLs that can be achieved under these conditions are given in Table 1. Other columns or element specific detectors may be used if the requirements of Section 9.2 are met.

10.1.2 Calibrate the system daily as described in Section 8.2.

10.1.3 Adjust the purge gas (nitrogen or helium) flow rate to about 40 mL/min. Attach the trap inlet to the purging device and open the syringe valve on the purging device.

10.2 SAMPLE INTRODUCTION AND PURGING

10.2.1 Remove the plungers from two 5-mL syringes and attach a closed syringe valve to each. Warm the sample to room temperature, open the sample (or standard) bottle, and carefully pour the sample into one of the syringe barrels to just short of overflowing. Replace the syringe plunger, invert the syringe, and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Add 10 μ L of the internal calibration standard to the sample through the syringe valve. Close the valve. Fill the second syringe in an identical manner from the same sample bottle. Reserve this second syringe for a reanalysis if necessary.

10.2.2 Attach the sample syringe valve to the syringe valve on the purging device. Be sure that the trap is cooler than 25°C, then open the sample syringe valve and inject the sample into the purging chamber. Close both valves and initiate purging. Purge the sample for 11.0 \pm 0.1 min at ambient temperature (Figures 1 and 3).

10.3 SAMPLE DESORPTION - After the 11-min purge, couple the trap to the chromatograph by switching the purge and trap system to the desorb mode (Figure 4), initiate the temperature program sequence of the gas chromatograph and start data acquisition. Introduce the trapped materials to the GC column by rapidly heating the trap to 180°C while backflushing the trap with an inert gas flow of 6 mL/min for 4.0 \pm 0.1 min. While the extracted sample is being introduced into the gas chromatograph, empty the purging device using the sample syringe and wash the chamber with two 5-mL flushes of reagent water. After the purging device has been emptied, leave the syringe valve open to allow the purge gas to vent through the sample introduction needle.

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10.4 TRAP RECONDITIONING - After desorbing the sample for four min, recondition the trap by returning the purge and trap system to the purge mode. Wait 15 s, then close the syringe valve on the purging device to begin gas flow through the trap. Maintain the trap temperature at 180°C. After approximately seven min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.

11. CALCULATIONS

11.1 Identify each analyte in the sample chromatogram by comparing the retention time of the suspect peak to retention times generated by the calibration standards and the laboratory quality control standard (Sect. 8.2.2) on the appropriate detector. When applicable, determine the relative response of the alternate detector to the analyte. The relative response should agree to within 20% of relative response determined from standards.

11.2 When both detectors respond to an analyte, quantitation is usually performed on the detector which exhibits the greater response. However, in cases where greater specificity or precision would result, the analyst may choose the alternate detector.

11.3 Determine the concentration of the unknowns by using the calibration curve or by comparing the peak height or area of the unknowns to the peak height or area of the standards as follows:

$$C_u = \frac{A_u}{A_{is}} \times \frac{C_{is}}{RF} \quad \text{Equation 3}$$

where: C_u = Concentration of the analyte in sample, in $\mu\text{g/L}$
 C_{is} = Concentration of the internal standard, in $\mu\text{g/L}$
 A_u = Peak area of the analyte
 A_{is} = Peak area of the internal standard
 RF = Relative response factor

11.4 Report the results for the unknown samples in $\mu\text{g/L}$. Round off the results to the nearest 0.1 $\mu\text{g/L}$ or two significant figures.

12. ACCURACY AND PRECISION

12.1 This method was tested in a single laboratory using reagent water spiked at 10 $\mu\text{g/L}$ (8). Single laboratory precision and accuracy data for each detector are presented for the method analytes in Table 2.

12.2 Method detection limits for these analytes have been calculated from data collected by spiking reagent water at 0.1 $\mu\text{g/L}$. These data are presented in Table 1.

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Table 1. CHROMATOGRAPHIC RETENTION TIMES AND
METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC COMPOUNDS
ON PHOTOIONIZATION DETECTION (PID) AND
HALL ELECTROLYTIC CONDUCTIVITY DETECTOR (HECD) DETECTORS

Analyte	PID Ret. Time, ^a min.	HECD Ret. Time, min.	PID MDL, ug/L	HECD MDL ug/L
Dichlorodifluoromethane	- ^b	8.47		.05
Chloromethane	-	9.47		.03
Vinyl Chloride	9.88	9.93	0.02	.04
Bromomethane	-	11.95		1.1
Chloroethane	-	12.37		0.1
Trichlorofluoromethane	-	13.49		0.03
1,1-Dichloroethene	16.14	16.18	N.D. ^c	0.07
Methylene Chloride	-	18.39		0.02
trans-1,2-Dichloroethene	19.30	19.33	0.05	0.06
1,1-Dichloroethane	-	20.99		0.07
2,2-Dichloropropane	-	22.88		0.05
cis-1,2-Dichloroethane	23.11	23.14	0.02	0.01
Chloroform	-	23.64		0.02
Bromochloromethane	-	24.16		0.01
1,1,1-Trichloroethane	-	24.77		0.03
1,1-Dichloropropene	25.21	25.24	0.02	0.02
Carbon Tetrachloride	-	25.47		0.01
Benzene	26.10	-	0.009	
1,2-Dichloroethane	-	26.27		0.03
Trichloroethene	27.99	28.02	0.02	0.01
1,2-Dichloropropane	-	28.66		0.006
Bromodichloromethane	-	29.43		0.02
Dibromomethane	-	29.59		2.2
Toluene	31.95	-	0.01	
1,1,2-Trichloroethane	-	33.21		N.D.
Tetrachloroethene	33.88	33.90	0.05	0.04
1,3-Dichloropropane	-	34.00		0.03
Dibromochloromethane	-	34.73		0.03
1,2-Dibromoethane	-	35.34		0.8
Chlorobenzene	36.56	36.59	0.003	0.01
Ethyl benzene	36.72	-	0.005	
1,1,1,2-Tetrachloroethane	-	36.80		0.005
m-Xylene	36.98	-	0.01	
p-Xylene	36.98	-	0.01	
o-Xylene	38.39	-	0.02	
Styrene	38.57	-	0.01	
Isopropyl benzene	39.58	-	0.05	
Bromoform	-	39.75		1.6
1,1,2,2-Tetrachloroethane	-	40.35		0.01
1,2,3-Trichloropropane	-	40.81		0.4

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Table 1 (Continued)

Analyte	PID Ret. Time, ^a min.	HECD Ret. Time, min.	PID MDL, ug/L	HECD MDL ug/L
n-Propyl benzene	40.87	-	0.004	
Bromobenzene	40.99	41.03	0.006	0.03
1,3,5-Trimethyl benzene	41.41	-	0.004	
2-Chlorotoluene	41.41	41.45	N.D.	0.01
4-Chlorotoluene	41.60	41.63	0.02	0.01
tert-Butyl benzene	42.92	-	0.06	
1,2,4-Trimethylbenzene	42.71	-	0.05	
sec-Butylbenzene	43.31	-	0.02	
p-Isopropyltoluene	43.81	-	0.01	
1,3-Dichlorobenzene	44.08	44.11	0.02	0.02
1,4-Dichlorobenzene	44.43	44.47	0.007	0.01
n-Butylbenzene	45.20	-	0.02	
1,2-Dichlorobenzene	45.71	45.74	0.05	0.02
1,2-Dibromo-3-Chloropropane		48.57		3.0
1,2,4-Trichlorobenzene	51.43	51.46	0.02	0.03
Hexachlorobutadiene	51.92	51.96	0.06	0.02
Naphthalene	52.38	-	0.06	
1,2,3-Trichlorobenzene	53.34	53.37	N.D.	0.03
Internal Standards				
Fluorobenzene	26.84	-		
2-Bromo-1-chloropropane	-	33.08		

a. Retention times determined on 60m x 0.75mm ID VOCOL capillary column.
Program: Hold at 10°C for 8 mins, then program at 4°C/min to 180°C, and
hold until all expected compounds have eluted.

b. - Dash indicates detector does not respond.

c. N.D. = Not Determined

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Table 2. SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR
VOLATILE ORGANIC COMPOUNDS IN REAGENT WATER

Analyte	Photoionization Detector		Hall Electrolytic Conductivity Detector	
	Recovery, ^a %	Standard Deviation of Recovery	Recovery, ^a %	Standard Deviation of Recovery
Benzene	99	1.2	- ^b	-
Bromobenzene	99	1.7	97	2.7
Bromochloromethane	-	-	96	3.0
Bromodichloromethane	-	-	97	2.9
Bromoform	-	-	106	5.5
Bromomethane	-	-	97	3.7
n-Butylbenzene	100	4.4	-	-
sec-Butylbenzene	97	2.6	-	-
tert-Butylbenzene	98	2.3	-	-
Carbon tetrachloride	-	-	92	3.3
Chlorobenzene	100	1.0	103	3.7
Chloroethane	-	-	96	3.8
Chloroform	-	-	98	2.5
Chloromethane	-	-	96	8.9
2-Chlorotoluene	N.D. ^c	N.D.	97	2.6
4-Chlorotoluene	101	1.0	97	3.1
1,2-Dibromo-3-chloropropane	-	-	86	9.9
Dibromochloromethane	-	-	102	3.3
1,2-Dibromoethane	-	-	97	2.7
Dibromomethane	-	-	109	7.4
1,2-Dichlorobenzene	102	2.1	100	1.5
1,3-Dichlorobenzene	104	1.7	106	4.3
1,4-Dichlorobenzene	103	2.2	98	2.3
Dichlorodifluoromethane	-	-	89	5.9
1,1-Dichloroethane	-	-	100	5.7
1,2-Dichloroethane	-	-	100	3.8
1,1-Dichloroethene	100	2.4	103	2.9
cis-1,2 Dichloroethene	N.D.	N.D.	105	3.5
trans-1,2-Dichloroethene	93	3.7	99	3.7
1,2-Dichloropropane	-	-	103	3.8
1,3-Dichloropropane	-	-	100	3.4
2,2-Dichloropropane	-	-	105	3.6
1,1-Dichloropropene	103	3.6	103	3.4
Ethylbenzene	101	1.4	-	-
Hexachlorobutadiene	99	9.5	98	8.3
Isopropylbenzene	98	0.9	-	-
p-Isopropyltoluene	98	2.4	-	-

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Table 2. (Continued)

Analyte	Photoionization Detector		Hall Electrolytic Conductivity Detector	
	Recovery, ^a %	Standard Deviation of Recovery	Recovery, ^a %	Standard Deviation of Recovery
Methylene chloride	-	-	97	2.8
Naphthalene	102	6.3	-	-
n-Propylbenzene	103	2.0	-	-
Styrene	104	1.4	-	-
1,1,1,2-Tetrachloroethane	-	-	99	2.3
1,1,2,2-Tetrachloroethane	-	-	99	6.8
Tetrachloroethene	101	1.8	97	2.4
Toluene	99	0.8	-	-
1,2,3-Trichlorobenzene	106	1.9	98	3.1
1,2,4-Trichlorobenzene	104	2.2	102	2.1
1,1,1-Trichloroethane	-	-	104	3.4
1,1,2-Trichloroethane	-	-	109	6.2
Trichloroethene	100	0.78	96	3.5
Trichlorofluoromethane	-	-	96	3.4
1,2,3-Trichloropropane	-	-	99	2.3
1,2,4-Trimethylbenzene	99	1.2	-	-
1,3,5-Trimethylbenzene	101	1.4	-	-
Vinyl chloride	109	5.4	95	5.6
o-Xylene	99	0.8	-	-
m-Xylene	100	1.4	-	-
p-Xylene	99	0.9	-	-

a. Recoveries and standard deviations were determined from seven samples spiked at 10 µg/L of each analyte. Recoveries were determined by internal standard method. Internal standards were: Fluorobenzene for PID, 2-Bromo-1-chloropropane for HECD.

b. Detector does not respond.

c. N.D. = not determined.

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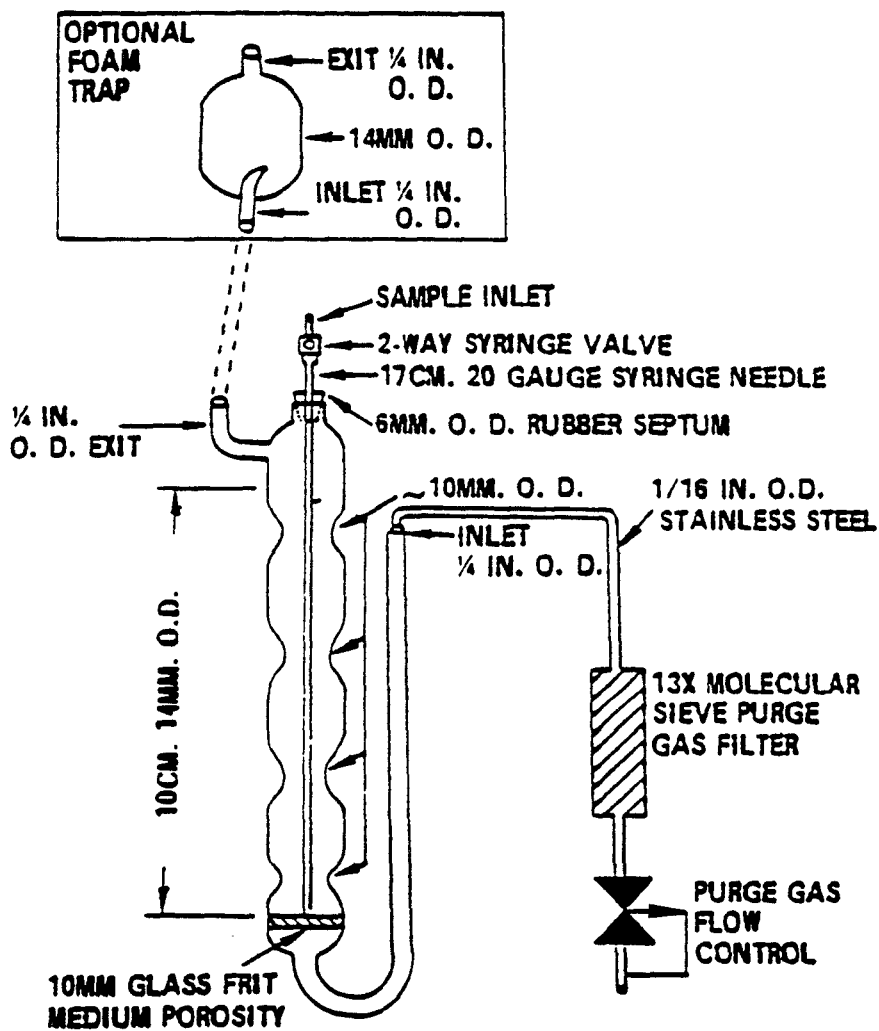


Figure 1. Purging device

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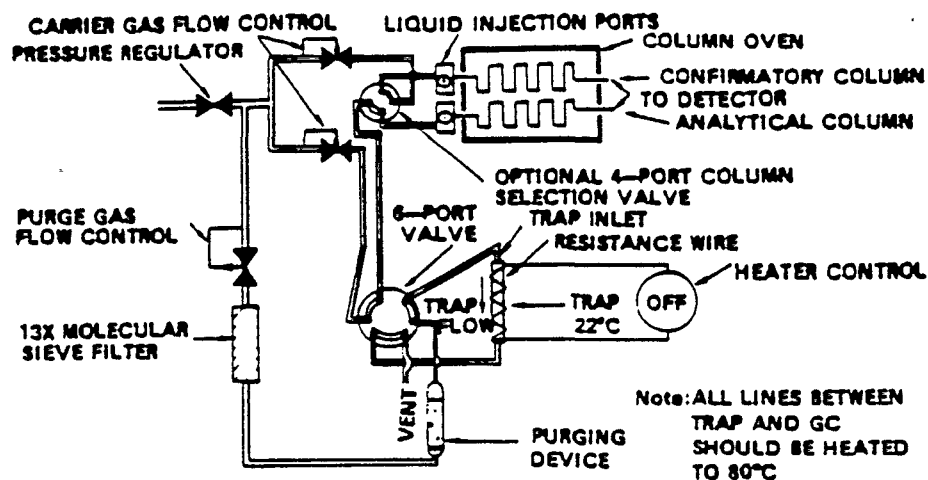


Figure 3. Purge and trap system - purge mode.

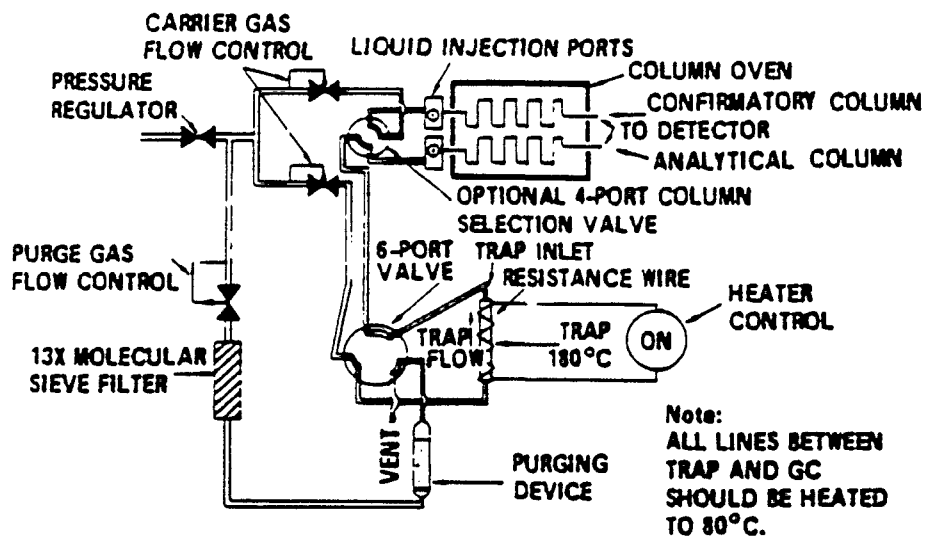


Figure 4. Schematic of purge and trap device - desorb mode

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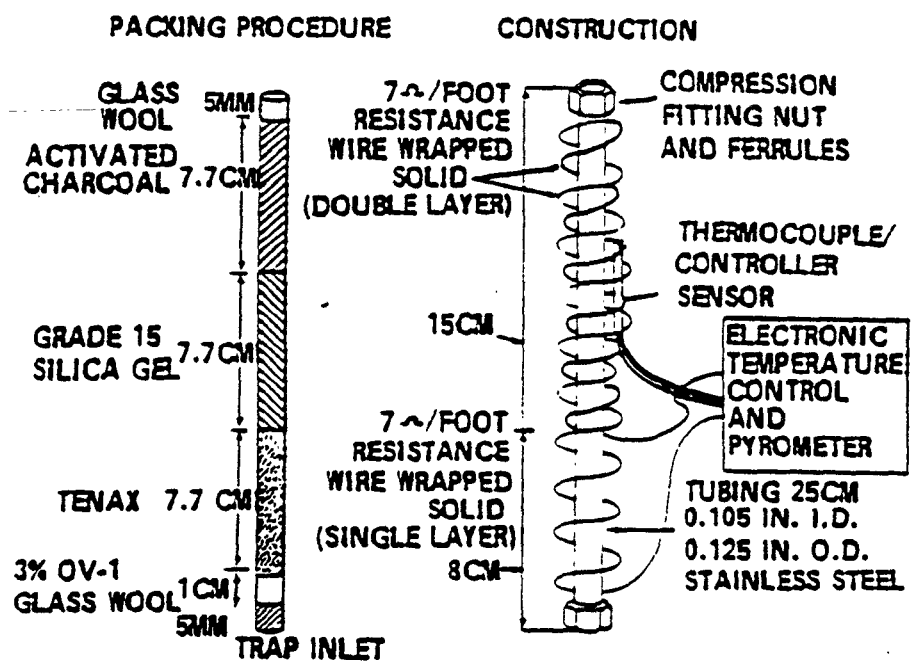


Figure 2. Trap packings and construction to include desorb capability

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COL 60 METER X 0.75 MM I.D. VOCOL CAPILLAR
PURGE AND TRAP VOC'S WITH HALL & PID IN SERIES

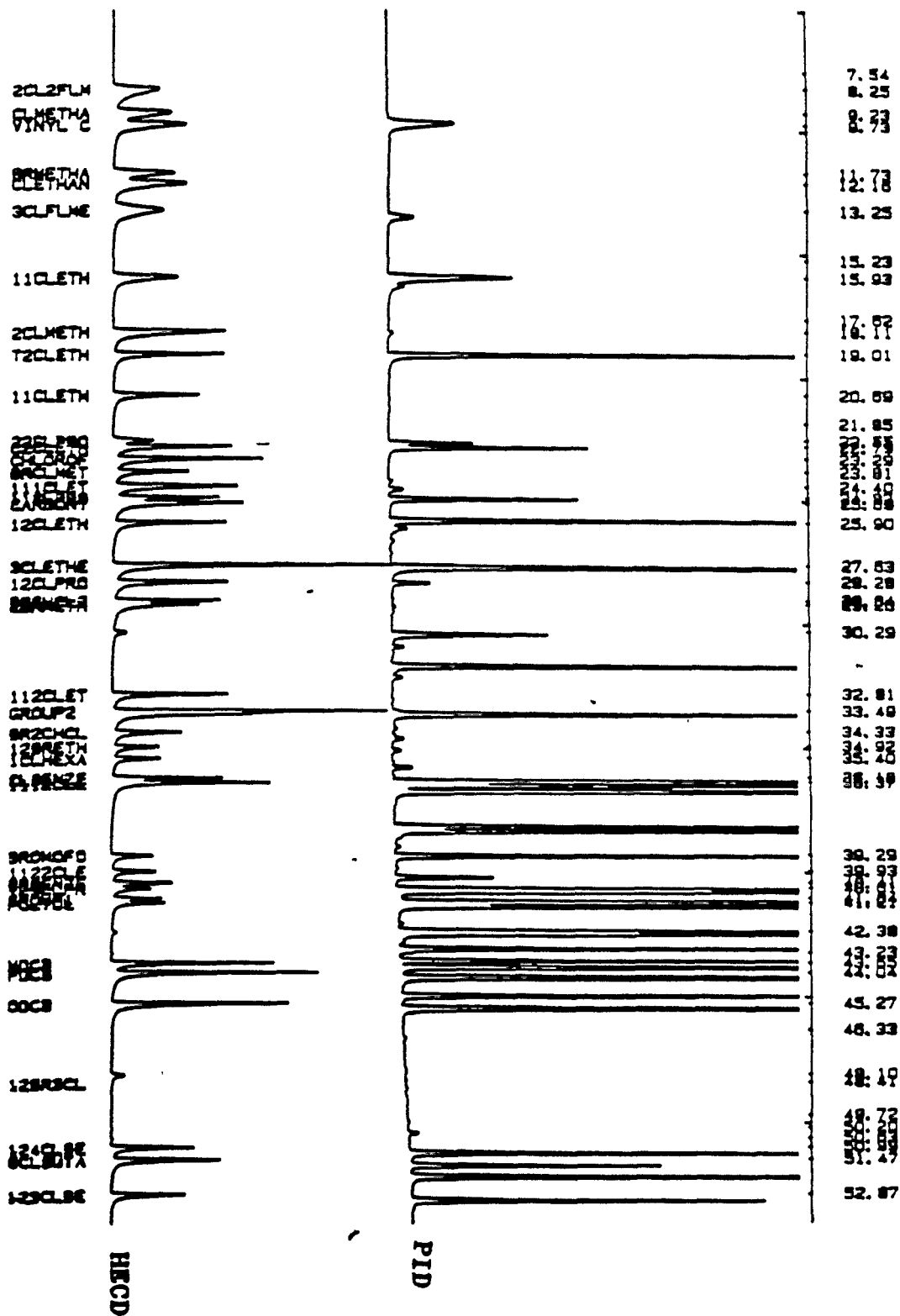


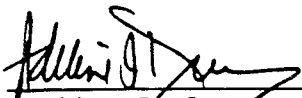
Figure 5. Gas chromatogram of volatile organics.

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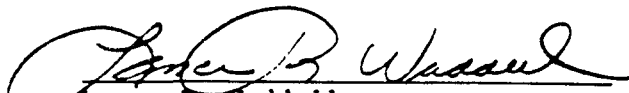
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LABORATORY - SPECIFIC ATTACHMENT

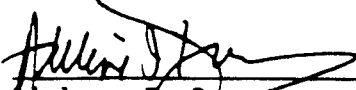
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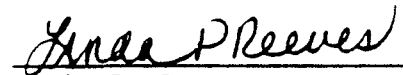

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SJ1.0 INTRODUCTION

Quality Assurance practices within International Technology Analytical Services (ITAS) laboratories are documented in the IT Analytical Services Quality Assurance Manual. The ITAS Manual was written for all analytical laboratories within International Technology Corporation. The purpose of the ITAS Manual is to provide uniform Quality Assurance practices among all IT laboratories. The manual recognizes that specific practices must be defined within each laboratory; therefore, the provision has been made for an individual laboratory to prepare an attachment to the ITAS manual which describes individual practice.

The IT laboratory located in San Jose, California has prepared a "laboratory-specific" attachment to the ITAS Quality Assurance Manual. As required by the ITAS Quality Assurance Program, this attachment is a supplement to the ITAS Quality Assurance Manual. In combination they provide the Quality Assurance Program for the local laboratory operations.

This laboratory-specific attachment is formatted in the same manner as the ITAS Quality Assurance Manual so that personnel in the San Jose laboratory operations can readily merge the requirements of the ITAS Manual and this document. Section headings for both documents are the same with the exception that this attachment uses the section prefix SJ so that attachment sections are clearly distinguished. The section prefix SC was used previously; SJ reflects the laboratory's name change from Santa Clara Valley to San Jose.

AR301051

The attachment is not meant to be a self-standing document; the ITAS Manual must be included with the attachment to fully describe the laboratory's Quality Assurance Program. Those sections not addressed in the attachment are adopted from the ITAS Quality Assurance Manual without change or modification. Thus, the attachment includes only those sections of the ITAS Quality Assurance Manual which are modified for full documentation of a specific activity, or which provide for the parallel implementation of Quality Assurance practices. The sections re-written and presented in the attachment are complete. In the event that the attachment describes an alternative means for implementation, when compared with the manner prescribed in the ITAS Quality Assurance Manual, the implementation described in this attachment shall prevail for San Jose laboratory operations. Review and approval of the attachment, as prescribed in this Manual is in accordance with the requirements of the ITAS Quality Assurance Program.

Table SJ1-1 lists the sections of the ITAS Quality Assurance Manual and Laboratory-Specific Attachment. If a section is not contained in the attachment, the corresponding section in the ITAS Manual is adopted without change or modification. Subsections are not listed in Table SJ1-1, but are fully identified in the Table of Contents.

AR301052

TABLE SJ1-1
CONTENTS OF LABORATORY-SPECIFIC ATTACHMENT
SAN JOSE LABORATORY

<u>Sections of ITAS Quality Assurance Manual</u>	<u>Topic</u>	<u>Attachment Sections</u>
1.0	Introduction	SJ1.0
2.0	Organization	SJ2.0
3.0	Standard Laboratory Practice	SJ3.0
4.0	Material Procurement and Control	SJ4.0
5.0	Sample Receipt and Initiation of Testing Program	SJ5.0
6.0	Calibration Practices	SJ6.0
7.0	Preventive Maintenance	SJ7.0
8.0	Analysis of Quality Control Samples	SJ8.0
9.0	Analytical Procedures	SJ9.0
10.0	Data Verification	SJ10.0
11.0	Data Reports	SJ11.0
12.0	Records Management	SJ12.0
13.0	Nonconformances and Corrective Action	SJ13.0
14.0	Quality Assurance/Quality Control Audits	SJ14.0
15.0	Quality Reports to Management	SJ15.0
16.0	Training	SJ16.0

AR301053

Within this manual, the terms Quality Assurance, Quality Control and Quality Assessment are defined as follows:

- . Quality Assurance- The overall controls imposed upon laboratory operations. Actions taken by personnel and the documentation of laboratory performance are included as specified in the Quality Assurance Program. Quality assurance includes quality control and quality assessment.
- . Quality Control- The daily, specific actions taken within the laboratory to control sample integrity, performance of analyses, data processing and record maintenance.
- . Quality Assessment- The system of activities taken to verify that the overall quality control job is being done effectively.

SJ1.3 DOCUMENT CONTROL, DISTRIBUTION, AND REVISION

The Quality Assurance Manual, Attachments, Manuals of Practice, Standard Operating Procedures and Project Specific Manuals are approved and controlled documents.

Approvals required before issue for the various documents follow:

- . ITAS Quality Assurance Manual
 - Senior Vice President, Environmental Projects Group
 - Vice President, IT Analytical Services
 - Director, Quality Assurance and Compliance
 - Corporate Director, Quality Assurance
- . Laboratory-Specific Attachment
 - General Manager, San Jose
 - Technical Director, San Jose
 - Director, Analytical Operations
 - Director, Quality Assurance and Compliance
 - Corporate Director, Quality Assurance

AR301054

- . Manuals of Practice
 - Vice President, IT Analytical Services
 - Director, Quality Assurance and Compliance, ITAS
- . Standard Operating Procedures
 - General Manager or Technical Director, San Jose
 - Quality Control Coordinator, Group Leader or Operations Manager, San Jose
 - Director, Quality Assurance and Compliance, ITAS
- . Project-Specific Manuals
 - General Manager, San Jose
 - Technical Director, San Jose
 - Quality Control Coordinator, San Jose
 - Project Manager (if analytical services are part of a broader-base project, and if required by contract)

Approval of these documents is denoted by a signature-and-date page in each document, which includes the required signatures above and the manual author. In addition, IT prepared documents are reviewed and approved by regulatory agencies, as appropriate.

In general, documents are distributed as needed to ITAS personnel. When the document is no longer needed or the copyholder leaves IT Corporation, it is returned. Copies distributed within IT and ITAS are numbered for document control, or are designated as uncontrolled documents. Distribution of the Quality Assurance Manual and Manuals of Practice is by the Director, Quality Assurance and Compliance, ITAS. Laboratory-Specific Attachments and Project-Specific Manuals (QAPP), and Standard Operating Procedures are distributed by the appropriate General or Laboratory Managers and Quality Control Coordinators.

AR301055

So that all Quality Assurance documents can be revised as necessary, distribution lists are maintained, as applicable, by the ITAS Quality Assurance Staff and laboratory Quality Control Coordinator. Before issue, revisions require the same approvals as the original document. Also, revisions are consecutively numbered and each page revised denotes the numbered revision. To facilitate revisions, each page of all manuals contains the following:

Title or Document Identification:
Section No. _____
Revision No. _____
Date: (of issue) _____
Page _____ of _____

Issuing a revision requires a revised signature page.

Revisions are issued to all holders of controlled copies. Each copyholder signs a revision receipt verifying that the revision has been received and properly placed in the document. The receipt is returned to the issuing manager.

Copies of Quality Assurance documents will be issued external to IT Corporation as required. These documents may be controlled (numbered) or uncontrolled (unnumbered) copies depending upon the reason for issue. Controlled copies will be revised in accordance with the above. In all cases, external parties will be asked to return Quality Assurance documents when the need for the document has ended. Exceptions to this will be if the external party requires a copy for record purposes.

AR301056

DOCUMENT

PURPOSE

QUALITY
ASSURANCE
MANUAL

- . Provides ITAS Quality Assurance policy
- . States responsibilities of laboratory staff
- . Describes basic Quality Control practices

LABORATORY-
SPECIFIC
ATTACHMENT

- . States supplementary implementing practices and services of the San Jose laboratory

MANUALS OF
PRACTICE

- . Detailed discussion of specific technical subjects
- . Provides detailed information related to technical topics discussed in the Quality Assurance Manual

STANDARD
OPERATING
PROCEDURES

- . Detailed description of specific tasks relating to laboratory operation and procedures

PROJECT-
SPECIFIC
MANUAL
(QAPP)

- . Prepared as required for specific contract
- . May supplement or change ITAS Quality Assurance practices for specific project
- . Takes precedence over other ITAS documents

FIGURE SJ1-1

ITAS-SJ QUALITY ASSURANCE DOCUMENTS

AR301057

SJ2.0 LABORATORY ORGANIZATION

This section discusses positions and quality-related responsibilities which the San Jose laboratory provides for the implementation of the Quality Assurance Program and completion of Quality Control activities. A discussion of the role of the IT Analytical Services Quality Assurance and Compliance Director is given in the ITAS Quality Assurance Manual.

SJ2.1 ITAS QUALITY-RELATED RESPONSIBILITIES WITHIN THE LABORATORY

An example organization chart is shown in Figure SJ2-1. The positions discussed below are part of the San Jose laboratory. The quality-related functions of these positions follow:

. General Manager

- Report directly to Vice President, IT Analytical Services, or Western Regional Director, Analytical Operations
- Implement the Quality Assurance Program within the laboratory
- Periodically determine effectiveness of the Quality Assurance Program in the laboratory
- Approve Laboratory-Specific Attachments to the Quality Assurance Manual, Project-Specific Manuals, Standard Operating Procedures and revisions
- Recommend to the Quality Assurance/Control Director, ITAS, changes in the Quality Assurance Program
- Maintain current laboratory organization chart.

. Technical Director

- Provide technical overview of laboratory activities

AR301058

- Serve as an "in-house" consultant for the applicability of general Quality Control practices to specific needs
- Evaluate analytical techniques, procedures, instrumentation and Quality Control methods, and provide recommendations to the Laboratory Manager
- Supervise the verification of software for data processing.
- Recommend standards for purchasing instrumentation, equipment, reagents, gases, and chemicals
- Supervise laboratory participation in inter-laboratory accreditation and proficiency programs
- Define the calibration program within the laboratory
- Define the instrument preventive maintenance program.
- Approve Laboratory-Specific Attachments to the Quality Assurance Manual, Project-Specific Manuals, Standard Operating Procedures and revisions.

. Quality Control Coordinator

- Prepare Quality Control standards, insert Quality Control samples into the laboratory sample stream, and evaluate results
- Review all statistical data to verify the laboratory is meeting stated Quality Control goals
- Recommend corrective actions for resolution of nonconformances
- Inform the General Manager, Technical Director and Operations Manager of serious nonconforming situations which cannot be immediately corrected

AR301059

- Report nonconformances to the Quality Assurance and Compliance Director if the situation is not corrected within the laboratory
- Stops production of data in a laboratory area where the review of quality control data or procedures shows significant problems
- Assist in the performance of Quality Assurance audits and perform Quality Control audits
- Establish and supervise the laboratory Quality Assurance training program
- Serve as the "focal point" for the reporting and disposition of all nonconformances
- Verifies corrective action and closes findings from Quality Assurance Audits
- Maintain current distribution lists for Laboratory-Specific Attachments, Project-Specific Manuals and Standard Operating Procedures
- Provide monthly reports to Quality Assurance and Compliance Director, ITAS
- Approve Standard Operating Procedures and revisions.

. Operations Manager

- Manage daily operations of the analytical laboratory
- Supervise Quality Control activities performed as part of routine analytical operations
- Review and approve report formats issued by the laboratory
- Train analysts in laboratory operations and analytical procedures
- Supervise sample storage facilities
- Supervise the preparation and maintenance of laboratory records
- Oversee instrument preventive maintenance schedule

AR301060

- Supervise the log-in of all samples received, completion of chain of custody, and maintenance of sample log books

- Approve Standard Operating Procedures and revisions.

. Project Manager

- Serve as in-house contact for client
- Review and approve all data summaries and reports issued by the laboratory
- Report out-of-control or nonconforming situations to Group Leader, Operations Manager and Quality Control Coordinator.

. Group Leaders

- Serve as the Lead Analyst within the group (group meaning organic analysis, inorganic analysis, special projects, etc.)
- Organize and schedule the analytical testing program with consideration for sample-holding times
- Supervise data verification procedures
- Review and approve all data summaries and reports issued by the laboratory
- Supervise instrument calibration and preventive maintenance programs
- Report out-of-control or nonconforming situations to Operations Manager and the Quality Control Coordinator
- Approve Standard Operating Procedures and revisions.

. Team Leaders

- Implement data verification procedures
- Assign analysts for data processing and validation activities

AR301061

- Review analytical data; prepare data reports and submit to Group Leader for final approval and reporting
- Evaluate instrument performance
- Report out-of-control or nonconforming situations to Group Leader and Quality Control Coordinator
- Supervise Quality Control activities performed as part of routine analytical operations.

. Analysts

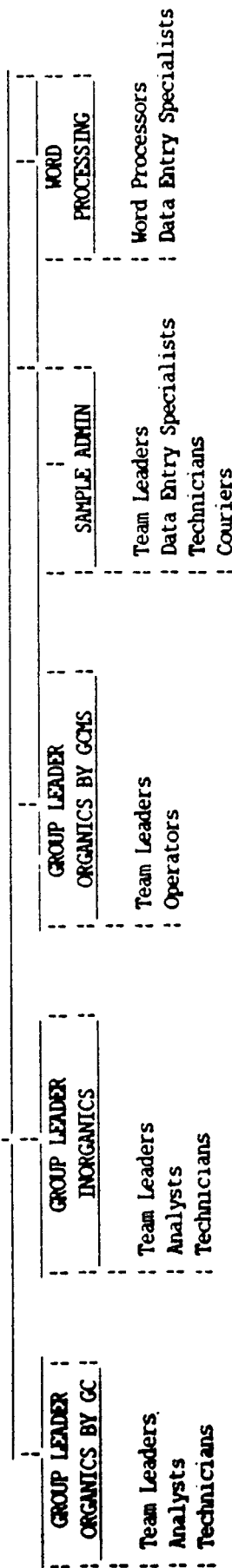
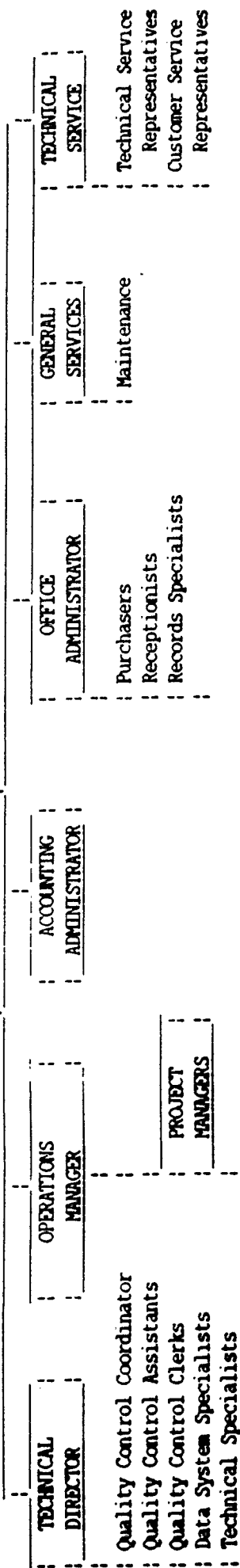
- Perform analytical procedures and data recording in accordance with accepted methods
- Perform and document calibration and preventive maintenance of instrumentation, as appropriate
- Perform data processing and validation
- Immediately report out-of-control situations, instrument malfunction, calibration failure, or other nonconformances to the Team Leader and Quality Control Coordinator.

. Lab Technicians

- Perform and document sample log-in
- Maintain sample storage facilities
- Perform and document sample disposal
- Assist in analytical procedures; perform procedures and data recording in accordance with accepted methods.

AR301062

GENERAL
MANAGER



Document: ITAS/SJ/QAM
 Section No. SJ2.0
 Revision No. 2
 Date: September 29, 1989
 Page 6 of 6

FIGURE SJ2-1

EXAMPLE ORGANIZATION CHART

AR301063

SJ3.0 STANDARD LABORATORY PRACTICE

SJ3.1 RECEIPT OF SAMPLES AND INITIATION OF TESTING PROGRAM

Upon receipt of samples in the laboratory, the following is done:

- . Samples are examined for damage, checked for proper preservatives and temperature
- . The Chain of Custody form is signed
- . Samples are placed in the proper storage environment
- . The testing program is defined by the Request for Analysis form or Purchase Order, or other appropriate means
- . Samples are logged into the laboratory sample stream
- . Appropriate laboratory personnel are notified of sample receipt.

SJ3.2 MATERIAL AND INSTRUMENT PREPARATION

Concurrent with the performance of analysis, auxiliary functions are performed to provide appropriate materials to the Analysts, and verify and maintain instrument performance.

Material Procurement and Control

- . Specifying grades of reagents, solvents, gases, and water used within the laboratory for specific analyses, and verifying adequacy of these materials
- . Specifying material composition and volumetric tolerance of laboratory vessels
- . Controlling reagents, solvents, etc., during storage
- . Cleaning protocol for laboratory vessels.

AR301064

Calibration

- . Scheduled comparison of instrument performance against national standards for instruments which measure physical parameters, such as mass, time, and temperature. This type of calibration is independent of use in specific analyses and projects.
- . Determination of instrument response to known chemical composition and concentration. Calibration may be part of daily instrument usage to define response for post analysis data processing of analytical raw data, or as response checks during or at the completion of an instrument run. This type of calibration may be independent of or part of the analysis for a specific project.

Preventive Maintenance

- . Servicing instruments on a scheduled basis to maintain performance
- . Maintaining a stock of instrument parts which are known to regularly degrade because of usage
- . Preparing log books so that the historical performance of an instrument can be assessed.

SJ3.3 ANALYTICAL PROCEDURES

The actual analysis of samples occurs during this function; however, for the analysis to be complete, several activities must be coincident:

- . Sample holding times and storage environment are reviewed by the Analyst or Team Leader to verify that sample integrity has been maintained
- . Analysis is performed in accordance with standard methods adopted by ITAS or as specified by the client. Analysis results include:

Prescribed daily instrument calibration or instrument tuning and documentation

Preparation and analysis of Quality Control samples and/or standards as part of the sample stream at the level of samples required.

AR301065

SJ3.7 REPORTING

- . Analytical data, and Quality Control data if appropriate, are summarized in presentation format
- . The data are reviewed by Group Leaders or representatives to verify that the objectives of the analysis have been met
- . After approval, the data are issued by the Project Manager, Group Leader, Operations Manager, Technical Director or General Manager.

SJ3.9 ANALYSIS FLOW CHART

An overview of laboratory operating functions is described in Table SJ3-1 and shown in Figure SJ3-1.

AR301066

TABLE SJ3-1
 ANALYSIS FLOW CHART
 SAN JOSE LABORATORY OPERATIONS

<u>STEP #</u>	<u>DESCRIPTION</u>	<u>RESPONSIBLE PERSONNEL</u>	<u>DOCUMENTS USED OR PRODUCED</u>
1	Receipt of samples at laboratory; inspection of samples; paperwork is verified for completeness and sample is placed in temporary storage.	Sample Receiving Personnel	Sample chain of custody, request for analysis form, letter of communication, communication memo and quote sheet, nonconformance memo, purchase orders, waybills.
2	Recording of sample information into the laboratory sample management system, preparation of project file for distribution of required information to personnel.	Sample Receiving Personnel	Items listed in Step 1 above, sample labels, job work order, project file, project index.
3	Storage of sample in proper area.	Sample Receiving Personnel	Job work order.
4	Project file is distributed to appropriate supervisor for processing.	Sample Receiving Personnel	Project file.
5	Project is assigned to analyst.	Group Leader or Team Leader	Project file, data summary form (manual or computer format).
6	Samples are collected from storage area for analysis.	Analyst or Lab Technician	Data summary form, job work order.

AR301067

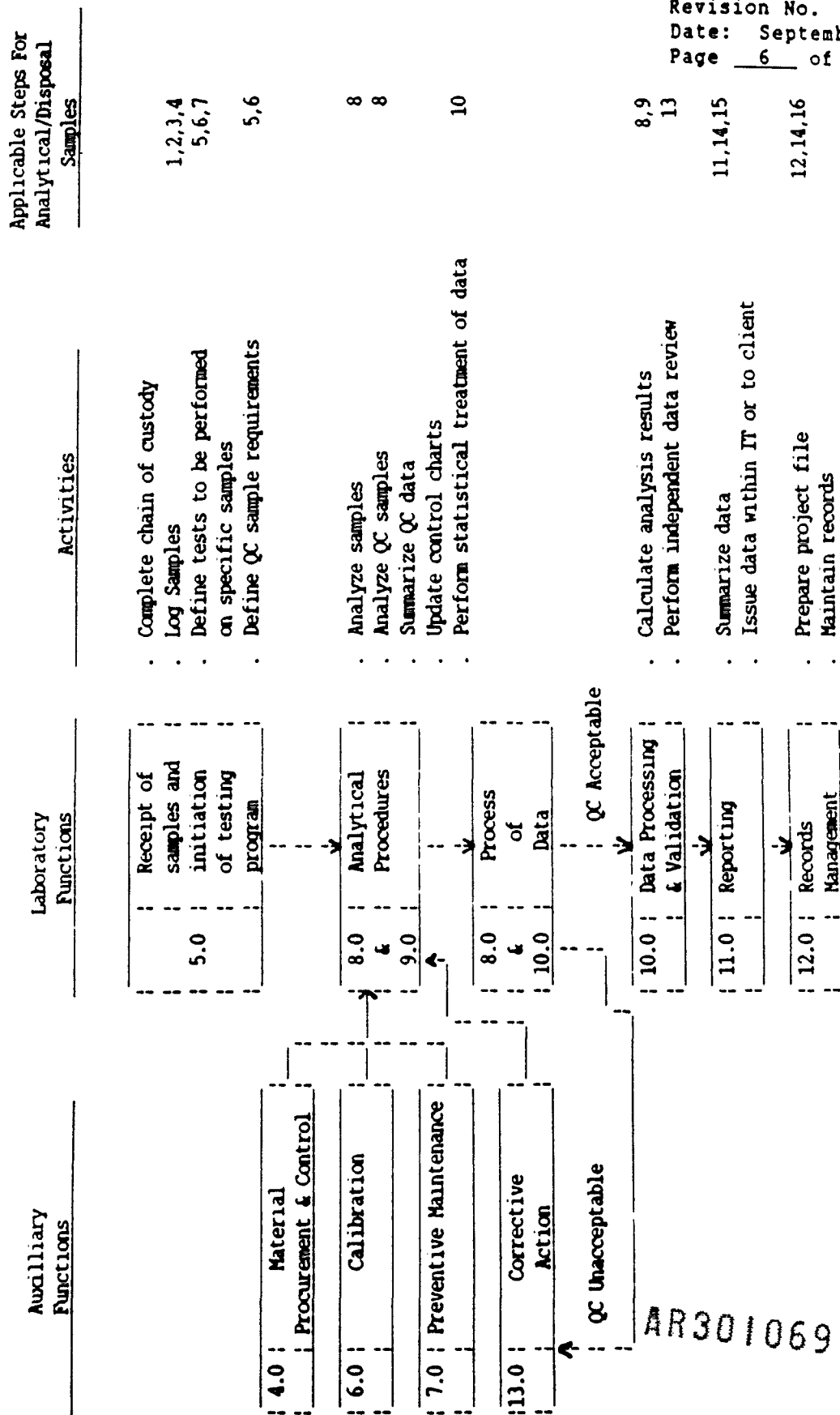
TABLE SJ 3-1
 ANALYSIS FLOW CHART
 SAN JOSE LABORATORY OPERATIONS
 (continued)

STEP #	DESCRIPTION	RESPONSIBLE PERSONNEL	DOCUMENTS USED OR PRODUCED
7	Analysis of sample.	Analyst or Lab Technician	Lab notebooks, bench sheets, data summary form, instrument log and data, calculation sheets, calibration records, QC sheets and charts, check-list.
8	Review of analytical data.	Second Analyst, Team Leader or Group Leader	Items listed in step 7 above.
9	Preparation of completed project file.	Analyst or Team Leader	Items listed in steps 1, 2 and 7 above.
10	Review of project file and preparation of report to client.	Team Leader or Group Leader	Items listed in step 9, request for report (manual or computer format).
11	Review and approval of report.	Project Manager, Group Leader, Operations Manager, Technical Director or General Manager	Items listed in step 10, final report
12	Storage of project and operational records.	Document Control Personnel	Items listed in step 11.
13	Disposal or archival storage of sample.	Lab Technician	Disposal and storage records.

AR301068

FIGURE SJ3-1

LABORATORY ANALYSIS FLOW CHART
SAN JOSE LABORATORY OPERATIONS



LEGEND: Applicable QA Manual section Step performed

AR301069

SJ4.0 MATERIAL PROCUREMENT AND CONTROL

The quality of reagents, solvents, gases, water and laboratory vessels used in analyses must be known so that their effect upon analytical results can be defined. Materials purchased by the San Jose Laboratory shall meet the requirements stated in the ITAS Quality Assurance Manual and this Laboratory-Specific Attachment or as denoted in specific analytical procedures.

The laboratory Quality Control Coordinator (QCC) has the overall control of materials to be used in the San Jose Laboratory. The QCC is responsible for defining the grades of materials to be purchased. Personnel use written records for purchasing materials. The receipt and distribution of materials is performed according to current standard operating procedures. Group Leaders are responsible for properly maintaining materials stored at the laboratory. They are also responsible for seeing that unsuitable or expired materials are removed from the laboratory for disposal.

SJ4.1.4 Water

Deionized water is monitored daily according to the current standard operating procedure. The specifications for ASTM Type II quality water shall be met: Maximum Electrical Conductivity at 25 degrees C of 1.0 umho/cm, or Minimum Electrical Resistivity at 25 degrees C of 1.0 Mcm/cm. AR301070

AR301070

Water which is used for any analyses (dilutions, preparation of reagent solutions, rinsing of glassware, etc.) must be free of interferences for the specific analysis performed.

SJ4.3 STORING AND MAINTAINING REAGENTS AND SOLVENTS

The following shall apply for storing and maintaining reagents and solvents:

- . Standard reagents and solvents are stored in appropriate cabinets and storage areas
- . Standard solutions are stored separately from samples to avoid possible cross contamination
- . Light-sensitive standard reagents or solvents are stored in a cool, dark place
- . Organic reagent standards are stored at or below 4 degrees C
- . Organic reference materials are stored in a freezer (at or below 0 degrees C)
- . Adsorbents for thin-layer and column chromatography are stored in the containers in which they are supplied, or according to the requirements of individual analytical methods
- . When fresh stock solutions are required, dilutions of the new standard are compared to the current standard or an independent Standard Reference Material to determine consistency and document traceability
- . Standards are not maintained longer than recommended by the manufacturer or as specified in the analytical method.

AR301071

SJ4.4 GLASSWARE CLEANING REQUIREMENTS

Methods of cleaning glassware are selected according to the substances that are to be removed and the analytical analysis required. Water-soluble substances can be washed out with hot or cold water, and if required, the vessel can be finally rinsed with deionized water. Other substances more difficult to remove may require the use of a detergent, cleaning solution, nitric acid, organic solvent or kiln-combustion. Details for cleaning glassware and containers specific for an analysis is given in current standard operating procedures.

AR301072

SJ5.0 SAMPLE RECEIPT, INITIATION OF TESTING

SJ5.2 CHAIN OF CUSTODY

An overriding consideration for resulting data is the ability to demonstrate that the samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Evidence of collection, shipment, laboratory receipt, laboratory custody and disposal must be documented to accomplish this. Documentation is accomplished through a Chain of Custody record that lists each sample and the individuals responsible for sample collection, shipment, and receipt. A sample is considered in custody if it is:

- . In a person's actual possession
- . In view after being in physical possession
- . Locked or sealed so that no one can tamper with it after having been in physical custody
- . In a secured area, restricted to authorized personnel.

Figure SJ5-1 is an example Chain of Custody form that is used by IT personnel in collecting and shipping samples. The San Jose laboratory should not accept samples collected by IT personnel for analysis without a correctly prepared Chain of Custody form.

The Chain of Custody form is signed by each individual who has the samples in their possession. Preparation of the Chain of Custody is as follows:

AR301073

- . The Chain of Custody record is initiated in the field by the person collecting the sample, for every sample. Every sample is assigned a unique identification number that is entered on the Chain of Custody form. Samples can be grouped for shipment and use a common form. The form of Figure SJ5-4 allows for ten samples. If more than ten samples are shipped in the same container, more than one Chain of Custody form is required.
- . The record is completed in the field to indicate project, sampling team, etc.
- . If the person collecting the sample does not transport the samples to the laboratory or deliver the sample containers for shipment, the first block for Relinquished By _____, Received By _____, is completed in the field.
- . The person transporting the samples to the laboratory or delivering them for shipment signs the record form as Relinquished By _____.
- . If the samples are shipped to the laboratory by commercial carrier or laboratory courier, the Chain of Custody form is sealed in a watertight container, placed in the shipping container, and the shipping container sealed prior to giving it to the carrier.
- . If the samples are directly transported to the laboratory, the Chain of Custody form is kept in possession of the person delivering the samples.
- . For samples shipped by commercial carrier, the waybill serves as an extension of the Chain of Custody record between the final field custodian and receipt in the laboratory.
- . For samples shipped by laboratory courier, the pick-up receipt (or equivalent) serves as an extension of the Chain of Custody record between the final custodian and receipt in the laboratory.

AR301074

- . Upon receipt in the laboratory, the Sample Custodian, or representative, opens the shipping containers, compares the contents with the Chain of Custody form, and signs and dates the record. Any discrepancies are noted on a nonconformance memo. If discrepancies occur, the field personnel are immediately notified.
- . Chain of Custody records are maintained with the records for a specific project, becoming part of the data package.

Multipart Chain of Custody forms may be used so that a copy remains with the person shipping the samples.

SJ5.3 LABORATORY SAMPLE RECEIPT

Samples are received in a designated area of the laboratory. Any samples that are damaged due to leaky or broken containers, have unusual odors or have violated the Sampling and Preservation Requirements of Table 5.1 of the ITAS QA Manual, are reported to the Project Manager or Customer Service Representative.

Samples are entered into the laboratory's Sample Management System using the procedure described in the laboratory's standard operating procedure. Each sample is assigned a unique laboratory number.

A work-order project file is initiated for each client's sample or group of samples having sequential laboratory numbers.

SJ5.4 LABORATORY STORAGE OF SAMPLES

AR301075

The primary considerations for sample storage are:

- . Maintenance of prescribed temperature, if required, which is typically 4 degrees C

- . Extracting and/or analyzing samples within the prescribed holding time for the parameters of interest.

The requirements of Table 5-1 (ITAS QA Manual) for temperatures and holding times will be used. Placing of samples in the proper storage environment is the responsibility of the sample receiving personnel, who should notify the Operations Manager, or responsible Group/Team Leaders if there are any samples which must be analyzed immediately because of holding-time requirements.

SJ5.5 INITIATION OF TESTING PROGRAM

A Request for Analyses form (Figures SJ5-2A, SJ5-2B) or letter of communication describing the required analytical testing should accompany all samples received at the laboratory. The form in Figure SJ5-2A is used for all IT projects and is recommended for use by other commercial clients. The form in Figure SJ5-2B contains an additional line for "Client Authorization". This form can be used for clients (other than IT) who do not have purchase orders or other authorization accompanying the request for analysis. If the analytical program for an IT project is not defined with the sample shipment, the Project Manager, Customer Service Representative or sample receiving personnel should notify the Project Manager responsible for the work for written definition of the analysis program. If the samples are external to IT Corporation, the Project Manager or Customer Service Representative should contact the client to determine the testing program and prepare communication notes for the project file.

AR301076

The analytical program will be entered into the laboratory's Sample Management System. An example work order form is shown in Figure SJ5-3.

The Group Leaders and Team Leaders are responsible for prioritizing samples on the basis of holding time and required reporting time into the laboratory sample stream.

SJ5.6 SAMPLE DISPOSAL

There are several possibilities for sample disposition:

- . The sample may be completely consumed during analysis
- . Samples may be returned to the client or location of sampling for disposal
- . The samples may be stored after analysis. Proper environmental control and holding time must be observed if reanalysis is anticipated. If reanalysis is not anticipated, environmental conditions for storage will not be observed. In general, ITAS will not maintain samples and extracts longer than sixty days beyond completion of analysis, unless otherwise specified
- . Disposal by the laboratory.

The Operations Manager or representative determines disposition of samples based on the current laboratory standard operating procedures, unless otherwise specified by the client.

AR301077



CHAIN-OF-CUSTODY RECORD

R/A Control No	A 80547
C/C Control No	

PROJECT NAME/NUMBER _____

LAB DESTINATION _____

SAMPLE TEAM MEMBERS

CARRIER/WAYBILL NO _____

[illegible]

Special Instructions:

Possible Sample Hazards

SIGNATURES (Name, Company, Date and Time)

1 Relinquished By: _____

Received By: _____

3 Relinquished By

Received by

2 Relinquished By: _____

Received By: _____

4 Relinquished By

Received By:

WHITE - To accompany samples
YELLOW - Field copy

FIGURE SJ5-1

EXAMPLE CHAIN OF CUSTODY FORM

Document: ITAS/SJ/QAM
Section No. SJ5.0
Revision No. 2
Date: September 29, 1989
Page 6 of 9



REQUEST FOR ANALYSIS

BILL TO

SEND LAB REPORT TO

PURCHASE ORDER NO

PROJECT CONTACT PHONE NO

B 84265

C/C Control No

[illegible]

TURNAROUND TIME REQUIRED
(Rush must be approved by the Project Manager)

Normal _____

Rush _____

(Subject to rush surcharge)

POSSIBLE HAZARD IDENTIFICATION (Please indicate if sample(s) are hazardous materials and/or suspected to contain high levels of hazardous substances)

Normal _____ Rush _____ (Subject to rush surcharge)

Neckband	Flammable	Skin Irritant	Highly Toxic	Other

(Please Specify)

SAMPLE DISPOSAL

Return to Client : --- Disposed by Lab

FOR LAB USE ONLY

Received By _____ Date/Time _____

WHITE - Original to accompany samples
YELLOW - Field copy

FIGURE SJ5-2A

EXAMPLE REQUEST FOR ANALYSIS FORM



INTERNATIONAL
TECHNOLOGY
CORPORATION
ITAS - SAN JOSE LAB
2055 Junction Ave.
San Jose, CA 95131
(408) 943-1540
FAX (408) 943-9332

REQUEST FOR ANALYSIS

WORK ORDER NO. _____

PROJECT NAME _____

PROJECT NUMBER _____

PROJECT MANAGER _____

BILL TO _____

DATE SAMPLES RECEIVED _____

LAB DESTINATION _____

LABORATORY CONTACT _____

SEND LAB REPORT TO _____

San Jose

DATE REPORT REQUIRED _____

DATE VERBAL RESULTS REQUIRED _____

PROJECT CONTACT _____

PURCHASE ORDER NO. _____

CLIENT AUTHORIZATION _____

PROJECT CONTACT PHONE NO. _____

SAMPLE FRACTION NO.	SAMPLE IDENTIFICATION	SAMPLE TYPE	DATE COLLECTED	SAMPLE VOLUME	PRESER- VATIVE	REQUIRED TESTING PROGRAM	CONDITION UPON RECEIPT
R301080							

SPECIAL INSTRUCTIONS _____

TURNAROUND TIME REQUIRED: Normal _____ Rush _____ (Subject to rush surcharge)

POSSIBLE HAZARD IDENTIFICATION: (Please indicate if sample(s) are hazardous materials and/or suspected to contain high levels of hazardous substances.)

Nonhazardous _____ Flammable _____ Skin Irritant _____ Highly Toxic _____ Other _____ (Please Specify)

SAMPLE DISPOSAL: (Please indicate disposition of sample following analysis. Lab will charge for packing, shipping and disposal.)

Return to Client _____

Disposal by Lab _____

FOR LAB USE ONLY

Received By _____

Date/Time _____

PAGE 1
 CLIENT: ITENG
 PROJ: 190067
 CON: FRED
 ITAS-San Jose
 WORK ORDER
 CAT: 190067
 04/10/89 00:07:51
 ORD # 53-04-073
 REV'D: 04/10/89 DUE: 04/14/89
 STAT: TRANSMITTED 04/10/89

COMPANY IT_Engineering
 FACIL Salinas
 REP Ed_Wing
 PHONE (408) 424-8040
 WORK ID waters_for_Firestone
 TAKEN by S.Hubenthal
 TRANS IT_courier/cool/ok
 TYPE waters
 INVOICE BY R
 % SURCHARGE
 % DISC: 00
 TEST/100
 # REP/INV 1 1
 SAMPLES 6
 KEEP FOR 30
 KEEP TO 04/14/89
 DASH
 DEPTS/ IESIS
 SAMPLE IDENTIFICATION
 JOB
 DE403 DW-1 (Outfall)
 DE403 DW-1 (Outfall)
 DE404 Chged. Trip Blank
 DE404 Chged. Trip Blank
 DE405 Vinion Dom #712
 DE405 Vinion Dom #712
 DE406 Vinion Dom #601
 DE406 Vinion Dom #601
 DE407 VINON-1
 DE407 VDCW-1
 DE408 VDCW-2
 DE408 VDCW-2

REPORT IT_Engineering
 TO 4585 Pacheco Blvd.
 Martinez, CA 94553
 AR30
 ATTN Maritess Pucan
 INVOICE IT_Engineering
 TO 4585 Pacheco Blvd.
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 ATTN Maritess Pucan
 PO# 190067
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TRANSMIT REPORTED INVOICED TOTAL \$
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WORK ORDER COMMENTS

... RUSH ...
 Analysis: VDA-10
 Project#: 190067 Project Name: Firestone

AR301081

SJ6.0 CALIBRATION PRACTICE

SJ6.1.1 Calibration Procedures

Written standard operating procedures and methods are used to calibrate equipment. These procedures and methods take into consideration the type of equipment, stability characteristics of the equipment, required accuracy, and the effect of operational error on the quantities measured. The procedures generally include:

- . Equipment to be calibrated
- . Reference standards used for calibration
- . Calibration technique and sequential actions
- . Acceptable performance tolerances
- . Frequency of calibration
- . Calibration documentation format.

SJ6.1.2 Equipment Identification

Equipment that is subject to calibration is uniquely identified so that calibration records can be designated with a specific instrument. Equipment identification is by a unique number assigned by the San Jose Laboratory.

SJ6.1.6 Calibration Records

Records are prepared and maintained for each piece of equipment subject to calibration. Records demonstrating accuracy of preparation, stability, and proof of continuity of reference standards are also maintained.

AR 301082

Records for periodically calibrated equipment include, as appropriate:

- . Identification number of equipment, type of equipment, and assigned unique equipment number
- . Calibration frequency and acceptable tolerances
- . Identification of calibration procedure used
- . Date calibration was performed
- . Identity of laboratory personnel and/or external agencies performing calibration
- . Reference standards used for calibration
- . Calibration data
- . Certificates or statements of calibration provided by manufacturers and external agencies, and traceability to national standards
- . Information regarding calibration acceptance or failure and any repair of failed equipment.

Records for periodically calibrated equipment are maintained in the Laboratory Operations Records as discussed in Section SJ12.0. Records for each instrument/equipment and physical reference standard are kept in a separate folder. The title sheet for each file is a summary of calibrations performed, such as shown in Figure 6 of the ITAS Quality Assurance Manual. It is recommended that an index precede the equipment files which lists in matrix form all equipment and physical standards, calibration frequency, and dates for upcoming calibration. The use of a calibration due date matrix provides ready reference so that calibration can be maintained by the responsible personnel.

AR301083

For instruments and equipment that are calibrated on an operational basis, calibration generally consists of determining instrumental response against compounds of known composition and concentration or the preparation of a standard response curve of the same compound at different concentrations. Records of these calibrations can be maintained in several ways:

- . The calibration data can be kept with analytical sample data
- . A log book can be prepared for each instrument which contains calibration data.

The former method provides response factor information, etc., directly with analytical data so that the analytical data can be readily processed and verified. Also, the raw data package is complete as a unit. However, if samples from several projects are processed together, the calibration data must be copied and included with each group of data.

The latter method provides an ongoing record of the calibration undertaken for a specific instrument; however, to process and verify the analytical data the log must be used in conjunction with the raw data.

SJ6.2 OPERATIONAL CALIBRATION

Operational calibration is performed as part of the analytical procedure and may have method-specific requirements. Since operational calibration is dependent upon the instrumentation within the laboratory, each calibration procedure is outlined in standard operating procedures.

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SJ6.2.2 Instrument Calibration Procedures

A summary of minimum operational calibration requirements is given in Table SJ6-1. Calibration is performed according to current standard operating procedures. The laboratory Quality Control Coordinator maintains a list of operational equipment requiring calibration.

SJ6.3 PERIODIC CALIBRATION

Periodic calibration for equipment such as balances, thermometers, and digital diluters is outlined in Table SJ6-2. The appropriate procedure is performed according to the laboratory's standard operating procedures. The laboratory Quality Control Coordinator maintains a list of equipment requiring periodic calibration.

SJ6.3.1 Balances

All balances are calibrated at least every three months using weights traceable to the National Institute of Standards and Technology (NIST). Calibration weights are Class S or better and need to be recertified every three years. If balances are calibrated by an external agency, verification of their weights are to be provided. Procedures used to perform the calibration are described in the laboratory's standard operating procedures.

SJ6.3.2 Thermometers

Working thermometers are compared with the reference thermometers every 12 months. Certified, or reference, thermometers are maintained for use in calibrating working thermometers.

AR301085

Reference thermometers are provided with NIST traceability for initial calibration and need to be recertified every three years with equipment directly traceable to the NIST. Procedures for calibrating thermometers are described in the laboratory's standard operating procedures.

AR301086

Table SJ6-1 SUMMARY OF MINIMUM OPERATIONAL CALIBRATION REQUIREMENTS

Instrument	Calibration Initial & Daily	Acceptance Limits and Corrective Actions
Gas Chromatograph	Initial: 3 levels & blank Daily: 1 check standard	Defined in SOP
Gas Chromatograph- Mass Spectrometry	Initial: 5 levels & blank Daily: 1 check standard	Defined in SOP
Liquid Chromatograph	3 levels & blank	Defined in SOP
UV-Visible Spectrophotometer	Initial: 3 levels & blank Daily: 1 check standard	Defined in SOP
Infrared Spectro- photometer	Initial: Wavelength calibration and 3 levels & blank Daily: Wavelength calibration and 1 check standard	
Atomic Absorption Spectrophotometer	Initial: 3 levels & blank Daily: 1 check standard every 10 samples	Defined in SOP
Inductively Coupled Plasma Emission Spectrophotometer	Initial: Manufacturer's Instructions Daily: Instrument check standard and cali- bration blank every 10 samples	Defined in SOP

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Table SJ6-1 SUMMARY OF MINIMUM OPERATIONAL CALIBRATION REQUIREMENTS (continued)

Instrument	Calibration Initial & Daily	Acceptance Limits and Corrective Actions
Total Organic Carbon	Daily: 3 levels & blank	Defined in SOP
Total Organic Halogen	Daily: 3 levels & blank	Defined in SOP
pH Meter	Daily: 3 levels	Defined in SOP

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Table SJ6-2 SUMMARY OF PERIODIC CALIBRATION REQUIREMENTS

Instrument	Calibration Initial & Daily	Acceptance Limits and Corrective Actions
Balances	Daily: Check with Class S weight Quarterly: Replicate weighings of Class S weights	Defined in SOP Defined in SOP
Class S weights	Every 3 years: Certification	Defined by NIST
Refrigerators	Daily: Measure temperature	Defined in SOP
Freezers	Daily: Measure temperature	Defined in SOP
Working thermometers	Yearly: Compare to NIST traceable thermometer	Defined in SOP
NBS Traceable Thermometer	Every 3 years: Certification	Defined by NIST
Conductivity Meter	Weekly: Check against standard solution	Defined in SOP
Digital Diluters, Pipettors	Quarterly: Gravimetric check	Defined in SOP

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AR301089

SJ7.0. PREVENTIVE MAINTENANCE

The preventive maintenance program at the San Jose Laboratory is an organized program of actions (such as equipment cleaning, lubricating, reconditioning, adjustment and/or testing) taken to maintain proper instrument and equipment performance and to prevent instruments and equipment from failing during use. This preventive maintenance program increases reliability of each measurement system.

The laboratory maintains a current inventory of instruments and equipment that are included in the program. The type and frequency of maintenance is scheduled on forms such as Figure SJ7-1.

The maintenance logs assigned to instruments and equipment are used to provide the necessary information for implementing repairs or service and to document any repairs, service or trouble-shooting performed. The logs provide a list of spare parts maintained by the laboratory, external service contracts, and items to be checked and/or serviced during maintenance and directions for performing maintenance (if external service is not provided). Entries to the log are made on forms such as Figure SJ7-2.

Table SJ7-1 gives examples of the requirements of the laboratory preventive maintenance program. A current schedule of preventive maintenance is maintained by the Quality Control Coordinator.

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Instrument/Equipment:

Serial Number: _____

[illegible]

Serial Number:

Page

[illegible]

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FIGURE SJ7-2

EXAMPLE PREVENTIVE MAINTENANCE RECORD

Table SJ7-1 PREVENTIVE MAINTENANCE

EQUIPMENT/ACTIVITY	ITEMS CHECKED/SERVICED	FREQUENCY
Atomic Absorption Spectrophotometer	PM Service	Semiannually
	Burner head	Each shift
	Electrical	Each shift
	Lamps	Each shift
	Nebulizer	Each shift
	Pump	Each shift
	Tygon tubing	Each shift
	Replace graphite tube	As needed, or 30-100 Samples
	Replace contact rings	As needed
	Replace quartz windows	As needed
	Clean optics around lamp area	Quarterly
	Vacuum/clean instrument	Monthly
Gas Chromatograph	PM Service	Annually
	EC (Ni-63) wipe test	Every 3 years
	Replace column packing	As needed
	Change column	As needed
	Replace septum	As needed
	Change gases	As needed
	Change fuses	As needed
	Clean or replace detector	As needed
	Change glass wool plug	As needed
	Clean insert	As needed
	Change carrier gas filter dryers	As needed
	Vacuum/clean instrument	Monthly
	Clean fan screens	Monthly
GC/MS	GC/MS maintenance is the same as GC with the following additions:	
	Mechanical pump oil	Quarterly
	Turbo pump oil	Quarterly
	Card cage air filter	Monthly

AR301093

Table SJ-7 PREVENTIVE MAINTENANCE (continued)

EQUIPMENT/ACTIVITY	ITEMS CHECKED/SERVICED	FREQUENCY
GC/MS (continued)	Source-clean ceramics, polish lenses	As needed
	Clean poles and ceramics on the poles	As needed
	Clean contacts on the component boards	As needed
	Vacuum the component boards	As needed
	Vacuum/clean instrument	Monthly
	Disk Drive	Semiannually (Serviced by Engineer) or as needed
	Printer	Quarterly
Purge and Trap Instrument	Replace Trap	As needed
	Replace Vessel	As needed
	Inspect unit	Monthly
	Vacuum/clean instrument	Monthly
Inductively Coupled Plasma Spectrophotometer	PM Service call	Semiannually
	Sample introduction system	Daily
	Clean, realign torch	As needed
	Clean nebulizer	As needed
	Clean mixing chamber	As needed
	Replace pump tubing	As needed
	Vacuum/clean instrument	Monthly
	Clean air filters	Monthly
Ion Chromatograph	Replace plunger seals	As needed
	Check plumbing	Daily
	Change column	As needed

AR301094

Table SJ7-1 PREVENTIVE MAINTENANCE (continued)

EQUIPMENT/ACTIVITY	ITEMS CHECKED/SERVICED	FREQUENCY
Ion Chromatograph (continued)	Change bed support	Replace when excessive back pressure
	Change fuses	As needed
	Change pump motor	As needed
	Clean check valve	As needed
	Clean, replace solvent reservoir filter	As needed
	Degas pump head	As needed
	Replace micro-membrane suppressor	As needed
	Replace ion exchange cartridge from suppressor system	Annually
	Change acid in regenerant reservoir	As needed
	Vacuum/clean instrument	Monthly
Liquid Chromatograph	Filter mobile phase	Each usage
	Run organic solvent through lines to disinfect	Each extended shut down
	Purge solvents	Each start up
	Replace guard column	As needed
	Change filter frits	Semi-annually
	Flush syringe with mobile phase	Each start up
	Clean, lubricate, inspect, adjust and test	Annual service
	Replace septum	As needed
	Change fuses	As needed
	Change columns	As needed
	Pump drives	Quarterly
	Wash dust filters	Quarterly
	Vacuum/clean instrument	Monthly

AR301095

Table SJ7-1 PREVENTIVE MAINTENANCE (continued)

EQUIPMENT/ACTIVITY	ITEMS CHECKED/SERVICED	FREQUENCY
UV-Visible Spectrophotometer	Clean cells Replace lamp Replace pump tubing Vacuum/clean instrument	Each usage As needed As needed Monthly
Infrared Spectrophotometer	Clean cells Inspect salt windows Vacuum/clean instrument	Each usage Quarterly Monthly
TOX Analyzer	Change electrolytes in titration cell Change H2SO4 scrubber Change/fill reference electrode Rebuild Agar bridge Repack pyrolysis tube	Daily Daily Daily Monthly As needed
TOC Analyzer	Change injection port septum IR Zero check/adjustment Check six port valve Check sample pump Change activated carbon scrubber Check digestion vessel Check gas permeation tube Check IR cell Check linearization	As needed Weekly Monthly Monthly Quarterly Quarterly Annually Annually Annually
Integrators, Printers	Check paper check print-ends or equivalent Vacuum/clean instrument	Daily Daily Monthly

AR301096

Table SJ7-1 PREVENTIVE MAINTENANCE (continued)

EQUIPMENT/ACTIVITY	ITEMS CHECKED/SERVICED	FREQUENCY
Computers	Vacuum/clean instrument	Monthly
pH Meter	Check electronics Change electrolyte	Daily Checked weekly, changed when low
Diluter and Pipettors	Clean and maintain area around units	Weekly
Freezers	Check and log temperature	Daily
Refrigerators	Check and log temperature	Daily
Walk-in refrigerator	Check and log temperature	Daily
Vacuum Pumps and Air Compressor	Check performance Check lubrication, belts, etc.	Weekly Monthly
Deionized Water Unit	Conductivity check of water Change ion exchange bed	Daily As needed

AR301097

SJ8.0 ANALYSIS OF QUALITY CONTROL SAMPLES AND STANDARDS

This section discusses samples which are routinely added to the normal laboratory sample stream to demonstrate that the laboratory is operating within prescribed requirements for accuracy and precision. Quality control samples are of known content and concentration (with the exception of trip blanks) so that accuracy and precision can be determined and control charts can be prepared. Evaluation of these data are discussed in Section SJ10.1. A summary of the frequency of laboratory QC samples and standards is detailed in the current standard operating procedures.

Table SJ8.1 summarizes the quality control samples and standards which are analyzed at the ITAS-San Jose Laboratory. Included in the table are:

- . Type of sample
- . Purpose of the sample
- . Frequency with which the sample is to be analyzed within the normal sample stream
- . Applicability of the sample to organic or inorganic analyses, with a citation if the sample is for GC/MS analysis only
- . Whether the sample is used for the statistical evaluation of accuracy and/or precision
- . Person responsible for introducing the quality control sample into the sample stream. If the sample is introduced by the Quality Control Coordinator, the content and/or concentration of the sample and its occurrence maybe unknown to the Analyst. Samples unknown to the analyst provide independent verification of laboratory operation.

AR301098

Following is a discussion of the major types of laboratory quality control samples. QC samples will be analyzed as recommended in the lab standard operating procedures unless analytical procedures prescribe specific sample analysis. If the procedure is specific, the procedural requirements will be met. A discussion of the ITAS interlaboratory testing program is given in the ITAS QAM.

SJ8.1 TYPES OF QUALITY CONTROL SAMPLES

SJ8.1.1 Trip Blank Analyses

Volatile organics samples are susceptible to contamination by diffusion of organic contaminants through the teflon-faced silicone rubber septum of the sample vial; therefore, trip blanks will be analyzed to monitor for possible sample contamination during shipment. Trip blanks will be prepared by filling two VOA vials with organic-free water and shipping the blanks with the field kit. Trip blanks accompany the sample bottles through collection and shipment to the laboratory and are stored with the samples. If the samples show the presence of analytes the trip blanks are always analyzed. If the trip blanks indicate possible contamination of the samples, depending upon the nature and extent of the contamination, the detection limit of associated samples may be raised, the samples may be corrected for the trip blank concentration or the sources resampled. Results of trip blank analyses should be maintained with the corresponding sample analytical data.

AR301099

SJ8.1.2 Field Blank Analyses

A field blank is a volume of water (or soil) that is provided by the sample collectors to demonstrate the absence of contamination during sampling. Contaminant-free water or contaminant-free solid material (lab sand, etc.) is placed into sample containers by the sample collection crew, packaged, and shipped with the other field samples. If analysis of the field blanks indicates possible contamination of the samples, depending upon the nature and extent of the contamination, the samples may be corrected for the field blank concentration or the sources resampled. Contamination sources to be checked include: Containers; sample storage facilities; field handling procedures; sampling tools (also see rinsate blanks). Results should be maintained with the corresponding sample analytical data.

SJ8.1.3 Rinsate Blank Analyses

A rinsate blank is a volume of rinse solution (contaminant-free water or organic solvent) used to rinse a sampling tool which contacts multiple samples. The rinse solution is collected after the tool has collected a sample and has been cleaned, to demonstrate that there is no residual contamination remaining on the tool to carry over into the next sample. If the rinsate blank indicates possible contamination of the succeeding samples, the samples may be corrected for the rinsate blank concentration or the sources resampled. Results of rinsate blank analyses should be maintained with the corresponding sample analytical data.

AR301100

SJ8.1.4 Method Blank Analyses

A method blank should be performed each day samples are processed, when the lot number of a principle reagent is changed, and at the rate of one out of twenty samples. Analysis of the blank verifies that method interferences caused by contaminants in solvents, reagents, glassware and other sample processing hardware are known and minimized. Optimally, a method blank should be free of those parameters being tested, but at most should contain no greater than the method detection limit for any parameter. Results of method blank analyses should be maintained with the corresponding analytical data.

SJ8.1.5 Reagent Blank Analyses

A reagent blank should be performed using materials which will be added to client samples during preparation. The purpose of the blank is to determine the degree of purity of the reagent so as not to allow a contaminated portion to affect the quality of the analysis. If a reagent blank is carried through the analytical procedure it becomes a method blank. Results of reagent blank analyses should be maintained with the corresponding analytical data.

SJ8.1.6 Bottle Blank Analyses

Standard operating procedures describe how laboratory-prepared sample containers are tested to verify that the glassware cleaning procedure is performed acceptably. Parameters of concern for the particular container are tested (metals for plastic containers, organics for glass, etc.). Results of the tests from each batch are filed according to the current standard operating procedures. Records of traceability of these containers through projects are maintained according to the current standard operating procedures.

AR301101

Vendors must supply certificate of cleanliness for purchased, pre-cleaned, containers, and these records are also maintained in the laboratory performance records.

SJ8.1.7 Duplicate Sample Analyses

Duplicate analyses are performed to evaluate the precision of an analysis. Results of the duplicate analyses are used to determine the relative percent difference between replicate samples. Criteria for evaluating duplicate sample results are provided in Section SJ10.1. Duplicate analysis results are summarized on the quality control data summary forms.

SJ8.1.9 Check Standard Analyses

Because standards and calibration curves are subject to change and can vary from day to day, a midpoint standard or check standard is analyzed to verify the standard curve and may serve in some cases as sufficient for calibration. Check standard analyses results are documented according to the current standard operating procedures.

SJ8.1.10 Surrogate Standard Analyses

Surrogate standard determinations should be performed on all samples and blanks for GC/MS analyses and for certain GC analyses. All samples and blanks are fortified with surrogate spiking compounds before purging or extraction to monitor preparation and analysis of samples. Recoveries should meet EPA acceptance criteria which are established as laboratory results become available. Surrogate standard data should be summarized and documented according to the current standard operating procedures.

SJ8.1.11 Laboratory Matrix Spike Analyses

To evaluate the effect of the sample matrix upon analytical methodology, a separate aliquot sample is spiked with the analyte of interest and analyzed with the sample. The percent recovery for the respective compound is then calculated. If the percent recovery falls outside established control limits, the data is evaluated and the matrix spike reanalyzed. Matrix spike results are summarized on the quality control data summary forms and plotted on recovery charts. This type of matrix spike does not necessarily reflect the behavior of the field-collected target analyte, especially if the target analyte is not stable during shipping or storage.

SJ8.1.12 Laboratory Duplicate Matrix Spike Analyses

To simultaneously evaluate the effect of the sample matrix upon analytical methodology and the precision of the method, separate sample aliquots are spiked in duplicate with the analytes of interest and analyzed with the sample. The percent recovery for the respective compound is then calculated. If the percent recovery falls outside established control limits for both matrix spikes, the data should be evaluated and the matrix spikes reanalyzed. Duplicate matrix spike results are summarized on the quality control data summary forms. Results of the duplicate analyses are also used to determine the relative percent difference between replicate samples. RPD results are summarized on the quality control data summary forms.

AR301103

SJ8.1.13 Verification/Reference Standard Analyses

On a quarterly basis, the Quality Control Coordinator will introduce a group of prepared verification samples, or standard reference materials, into the analytical testing regime. The data are reported to and summarized by the Quality Control Coordinator. Results of these data are presented to laboratory management for review and corrective actions, if appropriate.

SJ8.1.14 Blank Spike Analyses (Reagent Spike Analyses)

A reagent spike is a volume of contaminant-free water for water samples, or contaminant-free solid matrix for soil/sediment samples which is spiked with parameters of interest and carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of spiked materials demonstrates that the laboratory techniques for this method are in control. This sample is recommended in conjunction with matrix spike/matrix spike duplicate (MS/MSD) samples on those sample matrices which are anticipated to cause analytical difficulties due to matrix interferences. If the MS/MSD pair shows poor recoveries due to interferences, yet the blank spike sample is acceptable, this is strong evidence that the method has been performed correctly by the laboratory for these samples, but matrix interferences have affected the results. Results of blank spike analyses should be maintained with the corresponding MS/MSD and sample analytical data.

AR301104

TABLE SJ8-1
 QUALITY CONTROL SAMPLES AND STANDARDS

TYPE	PURPOSE OF SAMPLE	MINIMUM FREQUENCY	APPLICABILITY		ACCURACY AND PRECISION APPLICATION	INTRODUCED BY:
			INORGANIC	ORGANIC		
Trip Blank	VOA vial filled with contaminant-free water and taken with field sample collection kit. Used to verify that contamination of VOA samples has not occurred due to shipment and sample containers being in field.	A set of trip blanks with every set of collection vials sent to field.	X		Accuracy	Laboratory or Client
Field Blank	A volume of contaminant- free collection media is added to the container to verify absence of field contamination. May be water, soil, or other material.	Specified in Project Work Plan	X		Accuracy	Field Sampler
AR30 Quasate Blank	Rinse of field sample collection equipment to verify cleanliness, eliminate carryover of contamination to later samples.	Specified in Project Work Plan	X		Accuracy	Field Sampler
Method Blank	The analysis is performed using only the reagents and solvents used in the method. Determines cumulative interference. If interference cannot be eliminated, it must be considered when computa- tions are performed.	As described in current SOPs; not less than 1 per 20 samples.	X		Accuracy	Analyst

TABLE SJ8-1
QUALITY CONTROL SAMPLES AND STANDARDS
(continued)

TYPE	PURPOSE OF SAMPLE	MINIMUM FREQUENCY	APPLICABILITY		ACCURACY AND PRECISION APPLICATION	INTRODUCED BY:
			INORGANIC	ORGANIC		
Reagent Blank	Determine the background of each reagent/solvent to be used in an analyses. Must use identical conditions to actual analyses including detection system. Background must not interfere with intended analyses.	Can be done as part of method blank or separately.	X	X	Accuracy	Analyst
Bottle Blank	Analysis of cleaned sample container, analyzed to demonstrate lack of contaminants.	As described in current SOPs.	X	X	Accuracy	As described current SOPs
Duplicate	An aliquot of a sample known to Analyst. Calculate Relative Percent Difference.	As described in current SOPs.	X	X	Precision	Analyst
Blind Duplicate	An aliquot of a sample introduced unknown to analyst at time of sample log in by QC Coordinator (known to QC Coordinator).	One assay per group (semi-volatiles, volatiles, metals, etc.) per month.	X	X	Precision	QC Coordinator
Check Standard	Analysis of standard with concentrations at mid-point or low end of standard curve to verify standard calibration curve.	As needed to verify standard curve; described in current SOPs.	X	X	Accuracy	Analyst

TABLE SJ8-1
QUALITY CONTROL SAMPLES AND STANDARDS
(continued)

TYPE	PURPOSE OF SAMPLE	MINIMUM FREQUENCY	APPLICABILITY		ACCURACY AND PRECISION APPLICATION	INTRODUCED BY
			INORGANIC	ORGANIC		
Surrogate Standards	For GC/MS and certain GC analysis the addition of non-priority pollutants as spikes in standards, method blanks and samples.	Standards, methods blanks, and samples as described in methods.	X		Accuracy	Analyst
Spiked Samples (Laboratory Matrix Spikes)	A known concentration of a specific parameter is added to an aliquot of a sample with the matrix of interest. Percent recovery is determined and spike is compared against an unspiked aliquot.	As described in SOPs; not less than 1 per 20 samples.	X	X	Accuracy	Analyst
Matrix Spike Duplicate	Percent recovery is determined and compared against matrix spiked sample.	As described in SOPs; not less than 1 per 20 samples.	X	X	Accuracy and Precision	Analyst
Standard Reference Materials	Standards prepared by recognized external agency and used to determine performance of entire system, instrumentation and Analysts.	Scheduled quarterly basis.	X	X	Accuracy	QC Coordinator
Blank Spike	Blank material spiked with parameters of interest. Recovery evaluated for proper execution of technique or analytical method.	As needed	X	X	Accuracy	Analyst

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TABLE SJ8-1
QUALITY CONTROL SAMPLES AND STANDARDS
(continued)

TYPE	PURPOSE OF SAMPLE	MINIMUM FREQUENCY	APPLICABILITY		ACCURACY AND PRECISION APPLICATION	INTRODUCED BY
			INORGANIC	ORGANIC		
Field Matrix Spike	Sample spiked in field to show target analyte stability after collection, shipment, storage, preparation, and analysis	5 percent recommended	X	X	Accuracy	Field Sampler
Analysis Matrix Spike	Sample or extract spiked just prior to analysis. Shows matrix effects during analysis. Also known as "Method of Standard Addition" spike	As specified in methods, or as needed	X		Accuracy	Analyst
Internal Standards	Nonpriority pollutant spiked into sample and method blank after extraction to monitor instrument performance and sensitivity should surrogate standards indicate a problem. Applicable to certain organic analyses only (e.g. GC/MS 624, 625 etc).	Each sample and standard		X	Accuracy	Analyst
Collocated samples	Independent samples collected to be equally representative of the variables of interest.	5 percent recommended	X	X	Precision	Field Sampler
Replicated sample	Collected sample that has been split into two or more parts in the field, sent to the same laboratory	5 percent recommended	X	X	Precision	Field Sampler

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TABLE SJ8-1
 QUALITY CONTROL SAMPLES AND STANDARDS

TYPE	PURPOSE OF SAMPLE	MINIMUM FREQUENCY	APPLICABILITY		PRECISION APPLICATION	INTRODUCED BY
			INORGANIC	ORGANIC		
Split sample	Sample split into two or more portions, and sent to several laboratories for analysis.	1 percent recommended	X	X	Precision	Field Sampler or QC Coordinator

AR301109

SJ9.0 ANALYTICAL PROCEDURES

Analytical procedures for the analysis of samples include the following:

- . Prescribed method for sample preparation, including observance of stated sample holding times (such as shown on Table 5.1 of the ITAS QA Manual) and necessary extractions, dilutions, etc.
- . Instrument standardization, including calibration and preventive maintenance
- . Analytical techniques to be used in processing the sample
- . Prescribed format on prepared data sheets for recording raw data which include:
 - Identification of project(s)
 - Identification of sample number(s)
 - Identification of Analyst
 - Identification of Analyst performing data validation
 - Dates for sample analysis and data validation
 - Raw data resulting from the analysis with appropriate calibration standards and blanks
- . Method for computation of analytical results, which can be included on the data sheet.

The analytical procedures for various matrices (waters, soils, sediments, agricultural crops, environmental wipes, oils and other miscellaneous liquids and solids) are described in laboratory method manuals and method files. The method manuals are used for frequent analyses and include but are not limited to background information, published methods, superseded methods and the operating method. For non-frequent analyses, method files include advisory procedures and background information.

AR301110

SJ9.1 ANALYTICAL METHODS

Whenever possible, the San Jose Laboratory uses analytical methods published by agencies such as the U.S. Environmental Protection Agency (USEPA), U.S. Food and Drug Administration (USFDA) and the National Institute for Occupational Safety and Health (NIOSH). In addition, other published and in-house methods are used, especially in the field of pesticide residue analyses. The analytical methods used in the laboratory are maintained by the Technical Director.

SJ9.2 DETECTION LIMITS

All analytical methodologies have an associated method detection limit below which an analyte present in the sample cannot be accurately measured. The detection limits are either those given in approved methods or are established by performing the procedure described in the current standard operating procedure.

SJ9.3 VARIANCE FROM ANALYTICAL METHODS

Project specific requirements or analytical updates may require modification to the laboratory's current operating methods. Whenever a method is modified, documentation of the change is provided in the appropriate records (i.e., method manuals, method file or project file). Such a modification must be approved by the signatures of the Technical Director, ~~and the Operations Manager~~ *and QC Coordinator.*

AR301111

SJ10.0 DATA VERIFICATION

SJ10.1 PROCESSING OF QUALITY CONTROL DATA

This section discusses the analytical treatment of the data resulting from the quality control samples discussed in Section SJ8.0.

SJ10.1.1 Specific Routine Procedures Used to Assess Data Precision and Accuracy

Following are the procedures recommended for evaluating the precision and accuracy of data generated at the San Jose Laboratory. Quality control sample analyses are performed as discussed in Section SJ8.0. The protocol used will be in accordance with specific analytical procedures if QC requirements are stated in the procedure.

- . A reagent and/or method blank is prepared and analyzed each day samples are processed and at the minimum rate of one per twenty samples.
- . Trip blanks are analyzed to determine possible sample contamination during storage and shipment to the laboratory. Trip blanks are applicable to volatile organics analysis (VOA) where volatile contaminants can be introduced from ambient air on site, during shipment, and in the laboratory. The trip blank is run when samples are positive or may be used as method blanks. If run as a method blank and is positive, a "true" method blank must also be run.
- . Field blanks are analyzed to determine possible sample contamination during collection.
- . Bottle blanks are prepared and analyzed to demonstrate that clean containers were shipped to the field for sample collection. If purchased, vendor certificates should be retained to document cleanliness and lot numbers.
- . Rinsate blanks from the field are analyzed to show sampling equipment was properly cleaned between samples.

AR301112

- . In general, a daily calibration curve consisting of at least three standards and a reagent blank is prepared for each parameter. If the standard curve is known to be stable, the standard curve can be verified daily by the analysis of a midpoint standard.
- . A minimum of one sample in every sample set of 20 samples is analyzed in duplicate, or as a matrix spike duplicate.
- . A minimum of one sample in every sample set of 20 samples is spiked and analyzed.
- . A blind duplicate, unknown to the Analyst, is introduced periodically by the Quality Control Coordinator. Blind duplicates are routinely used for the analysis of metals, water quality parameters, and organics analyses.
- . Standard Reference Materials (SRMs) are introduced periodically into the testing scheme as verification or reference standards, by the Quality Control Coordinator to evaluate the accuracy of standards, the testing procedure, and the analyst's performance.
- . A blank spike consisting of analyte-free water or a purified solid matrix spiked with the parameter of interest is analyzed. Blank spikes are used to show the analytical technique was in control, although matrix effects may have been present in associated matrix spike cycles.
- . If appropriate, every sample is spiked with surrogate standards prior to extractions and analysis for certain organics.
- . Internal standards are added to all samples or extracts for GC/MS analyses and for certain GC analyses.
- . A check standard or midpoint standard is run to verify the continued acceptability of a calibration curve.
- . Analysis matrix spikes (or Method of Standard Additions) of target analytes are added to samples or extracts just prior to analysis to give information on matrix effects encountered during analysis.
- . Field matrix spikes are sample portions spiked with target analytes spiked into samples in the field to provide data on stability.
- . Collocated samples provide information on precision of intralaboratory or interlaboratory analytical systems when sent to the same or different laboratories, respectively.

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- . A replicated or split sample provides information about precision depending on the location in the sampling and analysis process where the split occurs. An analysis replicate gives information about the analytical precision; a field replicated or field split sample provides precision information for all steps after sample collection.

When the analyses of a sample set are completed, the results will be reviewed, as described in the current standard operating procedures and evaluated to assess the validity of the data set. Review is based on the following criteria:

- . Reagent or Method Blank Evaluation

The reagent and/or method blank results are evaluated for high readings characteristic of background contamination. If high blank values are observed, laboratory glassware and reagents should be checked for contamination and the analysis halted until the system can be brought under control before further sample analysis proceeds. A high background is defined as a background value sufficient to result in a difference in the sample value, if not corrected greater than or equal to smallest significant digit known to be true. A reagent or method blank should contain no greater than the parameter detection limit for most parameters.

- . Field, Trip, Bottle, and Rinsate Blank Evaluation

Field blank results are evaluated for high readings similar to the reagent and/or method blanks described above. If high trip blank readings are encountered, the procedure for sample collection, shipment and laboratory analysis should be reviewed. If both the reagent and/or method blanks and the trip blanks exhibit significant background contamination, the source of contamination is probably within the laboratory. In the case of VOAs, ambient air in the laboratory and reagent should be checked as possible sources of contamination. High field blank readings for other parameters may be due to contaminated sample bottles (unless bottle blanks are clean) or cross-contamination between samples collected if the rinsate blank is contaminated.

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. Calibration Standard Evaluation

The daily calibration curve is evaluated to determine linearity through its full range, and that sample values are within the range defined by the low and high standards. If the curve is not linear, ($r \geq 0.995$), sample values must be corrected for nonlinearity by deriving sample concentrations from a graph or by using an appropriate algorithm to fit a non-linear curve to the standards.

. Duplicate Sample or Duplicate Matrix Spike

Duplicate sample or duplicate matrix spike analysis for the sample set is used to determine the precision of the analytical method for the sample matrix. The duplicate results are used to calculate the precision as defined by the relative percent difference (RPD). The precision value, RPD, should be plotted on control charts for the parameter determined. If the precision value exceeds the warning limit (Section SJ10.1.2.2) for the given parameter, the appropriate Group Leader, Operations Manager, or the Quality Control Coordinator is notified. If the precision value exceeds the control limit, the corrective action may include the re-analysis for the parameter(s) in question. Attainable precision limits are taken from published methods or are calculated based on actual data.

. Matrix Spike And Blank Spike Evaluations

The observed recovery of the spike versus the theoretical spike recovery is used to calculate accuracy as defined by the percent recovery. The accuracy value, the percent recovery, may be plotted on a control chart for the parameter determined. If the accuracy value exceeds the warning limit for the given parameter, the appropriate Group Leader, Operations Manager, or the Quality Control Coordinator is notified. If the accuracy value exceeds the control limit, the corrective action may include the re-analysis for the parameter(s) in question. If interferences are present in the samples spiked, a blank spike is used to demonstrate that the laboratory technique is in control. The results of matrix spike and matrix spike duplicate parts of samples are compared as duplicate samples described above (RPD).

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. QC Check, or Laboratory Control Standard

Prepared as blank spikes, these samples are used for initial proficiency demonstration of analysts, methods, and equipment. Material is obtained with known values; it may be analyzed without spiking as a Laboratory Control Standard (LCS), similar to a Reference Standard.

. Blind Duplicate Evaluation

The blind duplicate analysis is evaluated in the same manner as described above for the duplicate sample analysis and is treated as a duplicate result for purposes of evaluating the precision of the analytical method. This evaluation is performed independently by the Quality Control Coordinator.

. Reference Standard Evaluation

Standard Reference Materials analyses are compared with true values and acceptable ranges. Values outside the acceptable ranges require corrective action to determine the source of error and provide corrective action. Analytical data reporting should be halted pending this evaluation. Following correction of the problem, the Standard Reference Material should be reanalyzed.

. Check Standard Evaluation

The results of check standard analysis are compared with the original calibration curve, and the relative percent difference of the check standard is calculated to determine if the calibration system is in control. If correction is required, the check standard should be reanalyzed to demonstrate that the corrective action has been successful.

. Surrogate Standard Evaluation

The results of surrogate standard determinations are compared with the true values spiked into the sample matrix prior to extraction and analysis and the percent recoveries of the surrogate standards are determined. Percent recoveries attained shall be in accordance with current EPA recommendations/requirements or, laboratory-generated control limits.

. Analysis Matrix Spikes Evaluation

Suppression or enhancement of instrument signal levels is demonstrated when recoveries are lowered or raised, respectively. Matrix-suppressing agents may be added to the sample or the extract to reduce such effects in routine sample analysis when the evaluation indicates samples are affected in this manner. One to three levels of analysis

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matrix spike concentrations are used to determine the unaffected concentration level native to the sample. The observed recovery versus the theoretical recovery is used to calculate the accuracy as defined by the percent recovery. Percent recovery may be plotted on control charts. If the accuracy value exceeds the control limits for a given parameter, the appropriate Group Leader, Operation Manager, or Quality Control Coordinator is notified. The reason for nonconformance must be determined, and appropriate corrective actions taken. These may include re-analysis for the parameter(s) in question.

. Field Matrix Spike Evaluation

Target analytes spiked into a portion of a field sample are measured to determine the amounts recovered after collection, shipment, storage, preparation and analytical processes have acted upon the analytes. Decreased recoveries may be due to bacteriological action on the poorly preserved sample, to photodegradation, to leaks from improperly capped/sealed containers, or degradation during physical treatment for analysis such as evaporation of organic solvents to reduce the sample extract volumes. The observed recovery versus the theoretical recovery is used to calculate the accuracy as defined by the percent recovery. Percent recovery may be plotted on control charts. If the accuracy value exceeds the control limits for a given parameter, the appropriate Group Leader, Operation Manager, or Quality Control Coordinator is notified. The reason for nonconformance must be determined, and appropriate corrective actions taken. These may include reanalysis of the sample(s) for the parameter in question.

. Collocated Sample Evaluation

Two or more samples processed independently by different people at different times using different techniques, or combinations of these variables, will yield results which provide an estimate of long-term precision of measurement systems. Or, if processed identically together, short-term precision is estimated.

. Replicate Sample Evaluation

Replicate sample analysis for the sample set is used to determine the precision of the sampling and analytical method for the sample matrix. Field splits, provide precision information about all steps after collection; analytical splits, or laboratory duplicates, provide information about instrument precision. The replicate results are used to calculate precision as RPD. Precision values exceeding control limits require the

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appropriate Group Leader, Operations Manager, or Quality Control Coordinator to be notified. The reason for the nonconformance must be determined and corrective actions taken. Laboratory duplicate precision limits are specified by the Quality Control Coordinator and updated periodically.

SJ10.1.2 Statistical Evaluation of Quality Control (QC) Data

As part of the analytical quality control program, the San Jose Laboratory determine precision and accuracy for each parameter or class of parameter analyzed. Initially, when these data are compiled, the evaluation is applied over a broad concentration range. As more data are accumulated, precision and accuracy determinations will be updated and criteria developed to define precision and accuracy over specific concentration ranges.

SJ10.1.2.1 Evaluation of Data Using Control Charts

Control charts are established for all major analytical parameters. In general a minimum of twenty measurements of precision and accuracy are required before control limits can be established. Control limits of three standard deviations are used. Preparation of control charts is described in the current standard operating procedures.

SJ10.1.2.2 Evaluation of Analytical Precision

General Considerations

To determine the precision of the method and/or laboratory Analyst, a routine program of duplicate analyses is performed. The results of the duplicate analyses are used to calculate the relative percent difference (RPD), which is the governing quality control parameter for precision.

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The RPD for replicate analyses is defined as 100 times the absolute value of the difference (range) of each duplicate set, divided by the average value (mean) of the duplicate set. For duplicate results D_1 and D_2 , the RPD is calculated from Equation 10-1:

$$RPD\% = \frac{|D_1 - D_2|}{\frac{D_1 + D_2}{2}} \times 100\% \quad (10-1)$$

When the RPD is obtained for a least twenty duplicate pairs, the average RPD and the standard deviation are calculated using:

$$\bar{m} = \frac{\sum_{i=1}^n m_i}{n} \quad (10-2)$$

and

$$S_m = \sqrt{\frac{\sum_{i=1}^n (m_i - \bar{m})^2}{n-1}} \quad (10-3)$$

where

m = the RPD of a duplicate pair,

\bar{m} = the average of the relative percent difference determinations,

S_m = the standard deviation of the data set of RPD determinations, and

n = the number of RPD determinations.

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When constructing a control chart for a specific parameter, the Warning and Control Limits are then calculated from the following:

$$\begin{aligned}\text{Upper Control Limits} &= \bar{R} + 3 S_m \\ \text{Lower Control Limits} &= \bar{R} - 3 S_m \\ \text{Upper Warning Limits} &= \bar{R} + 2 S_m \\ \text{Lower Warning Limits} &= \bar{R} - 2 S_m\end{aligned}$$

A control chart is established by plotting the RPD of each duplicate pair on a graph generated as follows:

- . The mean of the RPD determinations for the original data set is established as a solid horizontal line on the Y axis of the graph.
- . The Upper Warning and Control Limits calculated above are plotted as dashed horizontal lines across the graph at their respective points on the Y axis above the mean of the RPD determinations.
- . The Lower Warning and Control Limits calculated above are plotted as dashed horizontal lines across the graph at their respective points on the Y axis below the mean of the RPD determinations.
- . The calculated RPD of each duplicate pair is plotted on the graph to determine whether the RPD is within the Warning and Control Limits of the Control Chart.
- . If an RPD result is outside the Control Limits, the Team Leader and QCC are notified by means of a Nonconformance Memo. The nonconformance is investigated and a determination made of the source. The completed nonconformance memo is copied and attached to The Daily Raw QC Data Sheet.

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SJ10.1.2.3 Evaluation of Analytical Accuracy

In addition to the evaluation of analytical precision, the laboratory evaluates accuracy.

When a program for evaluation of analytical accuracy is established, the evaluation is applied over the entire range of spiking concentrations. As more data are accumulated, the evaluation procedure may be refined to define the analytical accuracy of the method over specific concentration ranges.

To determine the accuracy of an analytical method and/or the laboratory Analyst, a periodic program of sample spiking is conducted. The results of sample spiking are used to calculate the quality control parameter for accuracy evaluation, the Percent Recovery (%R).

The %R is defined as 100 times the observed concentration, minus the sample concentration, divided by the true concentration of the spike.

$$\%R = \frac{O_i - O_s}{T_i} \times 100\% \quad (10-4)$$

where

%R = the Percent Recovery,
 O_i = the Observed Spiked Sample Concentration,
 O_s = the Sample Concentration, and
 T_i = the True Concentration of the Spike

An example calculation for True Concentration is given in Equation 10-5: AR301121

$$T_i = \frac{\text{Spike Concentration [c] (mg/L) x Volume of Spike (in mL)}}{\text{Volume of Sample [in mL] + Volume of Spike [in mL]}} \quad (10-5)$$

When the Percent Recovery is obtained for at least twenty spiked samples, the mean percent recovery and the standard deviation are calculated using the formulae:

$$\bar{\%R} = \frac{\sum_{i=1}^n \%R_i}{n} \quad (10-6)$$

and

$$S_R = \sqrt{\frac{\sum_{i=1}^n (\%R_i - \bar{\%R})^2}{n-1}} \quad (10-7)$$

where

$\bar{\%R}$ = the Mean Percent Recovery,

$\%R_i$ = the Percent Recovery of a Single Spiked Sample,

n = the number of results, and

S_R = the Standard Deviation of the data set of Percent Recovery determinations.

The Warning and Control Limits are then calculated from the following equations:

$$\begin{aligned} \text{Upper Control Limit} &= \bar{\%R} + 3 S_R \\ \text{Lower Control Limit} &= \bar{\%R} - 3 S_R \\ \text{Upper Warning Limit} &= \bar{\%R} + 2 S_R \\ \text{Lower Warning Limit} &= \bar{\%R} - 2 S_R \end{aligned}$$

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A control chart is generated by plotting the Percent Recovery data on a graph as follows:

- . The mean of the Percent Recovery determinations for the original data set is established as the midpoint in the form of a solid horizontal line on the Y axis of the graph.
- . The Upper Warning and Control Limits calculated above are plotted as dashed horizontal lines across the graph at their respective points on the Y axis above the mean of the Percent Recovery determinations.
- . The Lower Warning and Control Limits calculated above are plotted as dashed horizontal lines across the graph at their respective points on the Y axis below the mean of the Percent Recovery determinations.
- . The calculated Percent Recovery of each spiked sample is plotted on the graph to determine whether the Percent Recovery is within the Warning and Control Limits on the Control Chart.
- . If a Percent Recovery result is outside the Control Limits, the Team Leader and QCC are notified by means of a Nonconformance Memo. The nonconformance is investigated and a determination made of the source. The completed nonconformance memo is copied and attached to the Daily Raw QC Data Sheet.

Completed control charts and QC data sheets are filed by the Quality Control Coordinator; current charts and sheets are kept at the work station for use in decision-making and updating.

SJ10.2 DATA VALIDATION

Data validation (review) is the process whereby data are screened and accepted or rejected, based on a set of criteria. Data is provided by 23 trained analysts using approved methods and instrument systems in control.

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Data validation is a systematic procedure of reviewing a body of data against a set of criteria to verify its validity prior to its intended use. It is applied to a body of data after the fact, systematically and uniformly. It must be applied close to the origin of the data by an independent and objective reviewer.

Data validation begins with the processing of data and continues through review of the data and the reporting of analytical results. Data processing can be performed by the Analyst who obtained the data or another Analyst. Data review starts with an Analyst independent of the data acquisition and processing, or the Group or Team Leader, reviewing (validating) that data processing has been correctly performed. It continues through verifying that the reported analytical results correspond to the data acquired and processed. Checks are made for internal consistency, proper identification, transmittal errors, calculation errors and transcription errors. Final review of the data to be reported is by the Group Leader or representative. The procedure is outlined in Figure SJ10.2.

As stated, the first step in validation is data processing. In general, data will be processed by an Analyst in one of the following ways:

- . Manual computation of results directly on the data sheet or on calculation pages attached on the data sheets.
- . Input of raw data for computer processing.
- . Direct acquisition and processing of raw data by a computer.

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If data are manually processed by an Analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors, dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, calculations should be done neatly on calculation paper, chromatograms, spectra, etc and included with the data sheets. The Analyst shall sign (full signature) and date in ink each page of calculations, and notebook. Only one signature and date per strip of continuous data (eg. chromatograms) is required.

For data that are input by an Analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the series number and/or other information as needed. The samples analyzed shall be evident and the input signed and dated by the Analyst. A small number of computer calculations should be checked manually.

If data are directly acquired from instrumentation and processed, the Analyst shall verify that the following are correct: project and sample numbers, calibration constants and response factors, output parameters such as units, and numerical values used for detection limits (if a value is reported as "less than"). The Analyst shall sign and date the resulting output.

SJ10.2.1 Review of Data Processing

Following is a discussion of the method to be used for reviewing (checking) data processing. At least 20% of all data shall be checked in this manner, unless there is a different project specific requirement. If during the checking process errors are determined such that any final result is

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changed, checking will be completely (100 percent) performed for the data set. If 100% checking is performed, a note to that effect will be entered and signed on the checklist of calculations and documentation. The steps for review are as follows:

- . The Analyst performing the data processing shall give an Analyst, independent of the work, the data package. The package shall include, as appropriate, raw data, data input/output, calculations, sources for input parameters such as response factors, etc.
- . The independent analyst (checker) will review the data for:
 - Appropriateness of equations used
 - Correctness of numerical input
 - Numerical correctness of all calculations; this should be done by re-performing numerical computations
 - Correct interpretation of strip charts, etc.
- . All entries and calculations that the checker reviews by re-performing the calculations completely will be marked in red ink with a check mark or a circle (only data actually checked should be check-marked or circled). The checking process must be thorough enough to validate that the results are correct. If the checker disagrees with any part of the computations, the checker shall mark through the number with a single red line and place the revised number above it in red ink.
- . Any changes made by the checker will be back-checked by the originator. If the originator agrees with the change, the checker insures that the results are corrected where appropriate. If the originator disagrees, the originator and checker must resolve the difference so they agree with the result presented.

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- . The checker will sign or initial originals and date in red ink all unreviewed pages of the data package (except for groups of printout such as chromatograms). Signing or initialing and dating indicates that the reviewer agrees with the calculations and that changes made have been agreed to by the originator.
- . If the data have been processed by computer, the reviewer will check every input entry. Agreement should be indicated by a check mark for every line. If the checker disagrees with the input, the number should be marked thoroughly with a red line and the corrected number indicated above it in red ink. Corrections must be back-checked by the originator as discussed above.
- . If the input error is identified and the data have been processed, it will be necessary to reprocess the data. In this event, the checker shall mark in red ink the second set of input to indicate agreement with the input changes. The checker shall sign or initial and date in red ink the computer input to indicate agreement.
- . Raw data that are automatically acquired and processed do not require any validation at this point beyond that previously discussed.
- . The reviewed data are maintained as discussed in Section SJ12.0.

SJ10.2.2 Review of Data Reporting

Review of data reports is required to verify that information reported by the San Jose laboratory corresponds with processed analytical results. Review is only required of the data as it is presented for issuance. Intermediate steps performed after the processed data are checked to prepare the data report (such as data summaries) do not require validation. Referring to Figure SJ10-2, preparation of the report is the responsibility of the Project Manager or his representative. AR301127

After the data report is prepared (generally in tabular form), the reported results should be checked against the reviewed processed data so that transcription errors do not occur. The checking process follows:

- . Using a copy of the report, all data entries are checked. The checker can be the Project Manager or his representative. The checker is not required to be independent of the work because only the transcription from the reviewed data to the data report is being checked.
- . The copy of the data report should be checked so that the items cited for data presentation in Section SJ11.0 are complete and correct. As the reviewer checks the entries on the report, an ink check mark beside or circle around is made for each correct entry. Corrected entries are marked through with a single line and the correct entry provided in ink. The reviewer will indicate that corrections have been made in the report by placing a check mark or circle in ink by the correction in the revised corrected copy. The checker will place a red check mark at the bottom righthand corner of each page of the copy of the data report. The checker then signs and dates the first page of the copy of the data report.
- . The copy of the data report which is used as a checkprint should be maintained as a record to demonstrate the review.
- . If data printouts, such as chromatograms or GC/MS data processing, are included in the data report, review is not required for the data printout.
- . If computer output is used directly as the data report without further transcription, only the input requires review as discussed in Section SJ10.2.1.

After checking the copy of the data report it is signed for issue by the Project Manager, Group Leader, Operations Manager, Technical Director or General Manager.

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SJ10.2.3 Initial and Signature Identification

The Quality Control Coordinator will maintain a current list of employees with their actual initials and signatures so these can be positively identified.

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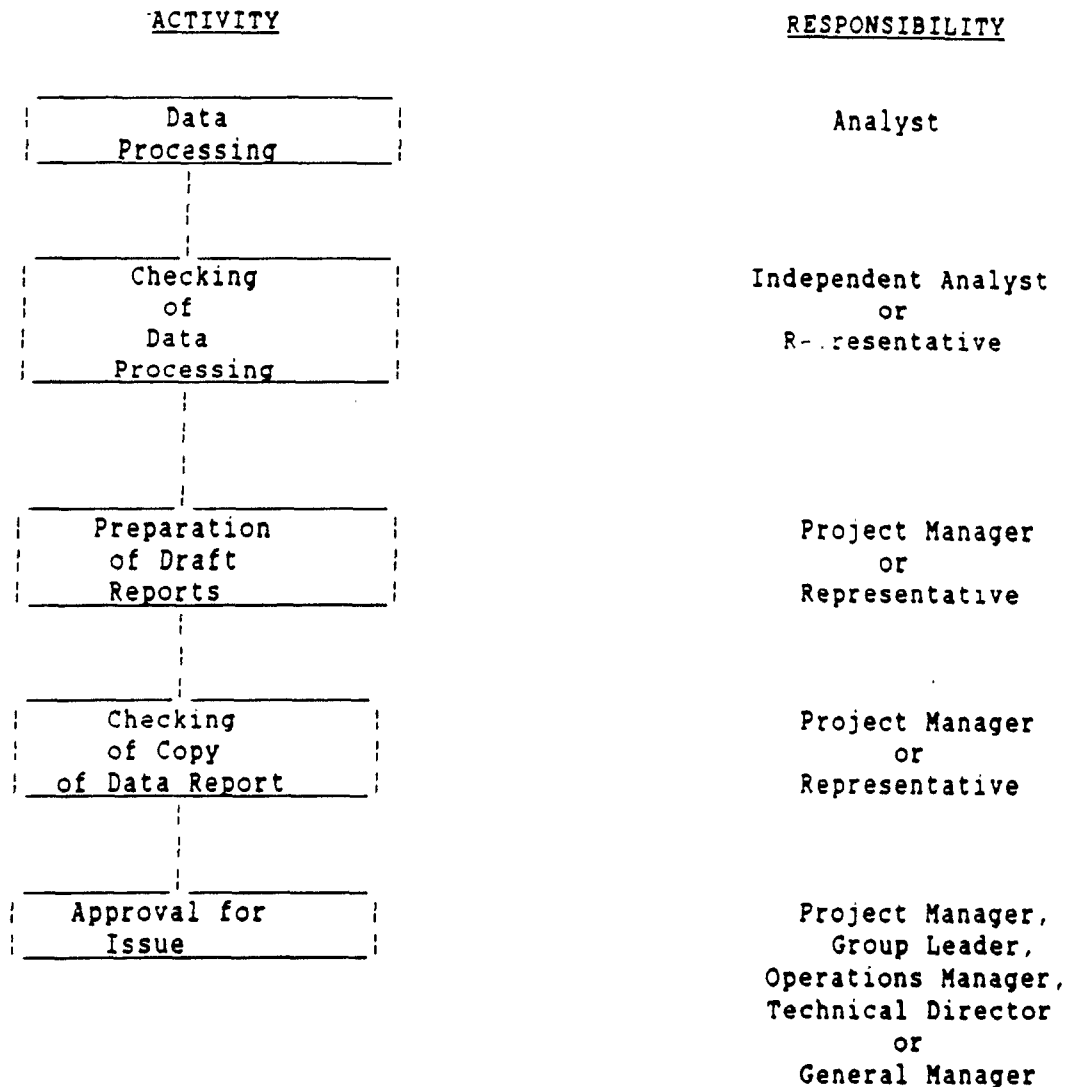


FIGURE SJ10-2
 DATA VALIDATION PROCESS

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SJ11.0 DATA REPORTS

Data Reports are dependent upon project needs, such as client or contract requirements. The following items are applicable to data presentation and the report format:

- . The final data presentation is checked in accordance with data verification requirements of Section SJ10.0 and approved by the Project Manager, Group Leader, Operations Manager, Technical Director or General Manager
- . Data are generally formatted as a Certificate of Analysis such as shown on Figure 11-1 of ITAS QA Manual
- . Explanatory text is included with the results
- . Data are presented in a tabular format whenever possible
- . Each page of data is identified with the date of issue, page number, client name and if appropriate, the project number and name
- . Data presentation includes:
 - Sample identification number used by the San Jose Laboratory and the sample identification provided to the laboratory
 - Matrix, chemical parameters analyzed, reported values, and units of measurement
 - Detection limit of the analytical procedure
 - Consistent use of significant figures, when reporting data for a sample set
 - Results of Quality Control sample analysis if requested as part of the project plan
 - Achieved accuracy, precision and completeness of data if requested as part of the project plan

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- Footnotes referenced to specific data if required to explain reported values
- . Written data is transmitted from the laboratory only by the Project Manager, Group Leader, Operations Manager, Technical Director or General Manager.

Any analytical results verbally communicated are considered preliminary until data are presented in hard copy.

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SJ12.0 RECORDS MANAGEMENT

SJ12.1 PROJECT RECORDS

Project files are maintained for each group of samples analyzed at the laboratory. The records are filed by client name, client project number and laboratory work order number. There are several categories of information within a project file.

Table SJ12.1 presents the categories used within a project file. It is not expected that all categories will be applicable for every project. However, in all project files, specific information will be filed in accordance with the category designation provided. Following is a brief discussion of additional documents and categories which are specific to the San Jose Laboratory. Details regarding how the documentation is compiled is given in the current standard operating procedure.

Project Index or Project Checklist

Each project file shall have an index or checklist that lists each record unit within the project file.

Figure SJ12-1 presents an example of a project index or checklist page.

Items are stored in the order they appear in the checklist.

A - Correspondence

All correspondence pertinent to the analytical program is maintained.

This includes letters to and from clients, internal memorandums, purchase

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orders, etc. Data reports and analysis requests are maintained in separate categories. The correspondence file is updated as necessary.

B - Chain of Custody

Chain of custody records maintained by the laboratory are filed for groups of samples as received. Copies of Chain of Custody records for samples sent to other laboratories are maintained so the custody chain is unbroken.

C - Field Records

All field records supplied by field personnel pertinent to the analytical program are maintained. This can include the results of field tests or logs of sample collection.

D - Request for Analysis

Analysis requests provided by field personnel are maintained in this file. Also, any changes or additions to the analytical program are documented in this file. If the analysis request is combined with the Chain of Custody form, reference to the Chain of Custody is noted on the project index or project checklist.

E - Calibration Records

In general, calibration records are maintained with laboratory operation records. However, if an analytical program requires calibration which is performed solely for a project, the records are maintained in this file. If calibration is performed as an integral part of the analytical process, the calibration records are maintained with the analytical data.

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F - Analytical Data

Analytical data files are complete for a group of samples in accordance with the needs of the project. The file contains a summary of the results and may reference the instrument series in the operation files or contain raw analytical data, processing of the data and/or data reduction, and data validation.

G - Quality Control Samples

If Quality Control samples, such as field blanks, are processed for a specific project, the data is maintained with the project file. The results of Quality Control samples processed on a general basis are included in the laboratory operations files. Statistical evaluation of Quality Control sample data for a project is also maintained in the operations file.

If Quality Control samples are processed as an integral part of a group of samples such that the data cannot be readily separated, the Quality Control sample data is stored with the analytical data.

H - Data Reports

Complete copies of all reports issued by the laboratory are kept in the project file. With the reports is the review copy checked to verify the data. If the report provides copies of data packages which are stored in Category F - Analytical Data, a memorandum can be attached to the report copy stating which specific files were included.

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I - Project-Specific Requirements

If a project requires analytical procedures other than those adopted in the ITAS Quality Assurance Program, the requirements are included in this file. Specific requirements may be due to government regulation, specific contracts, or project need. Changes from stated practice can be, for example, the frequency of QC sample analysis, test method, statistical data evaluation, and reporting format.

J - Nonconformances

Copies of nonconformances which are specific to a project are included in this file. Each nonconformance has a unique identifier which can be cross-referenced to the operations files to determine subsequent corrective actions.

K - QA Plans

Any specific Quality Assurance Plans, and revisions, which are prepared for a project, are stored in this file.

L - Miscellaneous

The miscellaneous file includes all records not applicable to the previous categories. Each distinct record(s) in this file is entered in the project index or project checklist.

M - Method Description

Description of all methods used are maintained by the laboratory. If a specific method is used for a project, the procedures are kept with the project file. Methods that are used on a general basis are included in the

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laboratory operations files. The index may indicate that the method reference can be found in the instrument series which is filed with the laboratory operation records.

N - Work Order Records

Internal tracking documents used by the laboratory (e.g., computer-generated records, etc.) are included as part of the traceability of the project samples through the laboratory.

SJ12.2 GENERAL LABORATORY OPERATIONS RECORDS

General laboratory records document overall laboratory performance and operation. These records are filed separately from project records and are maintained so they can be referenced to project records if necessary. Examples of general records pertinent to project records are instrument maintenance logs, calibration and computer software verifications.

There are two types of general laboratory records:

- . Documents which demonstrate laboratory performance
- . Reference documents for laboratory operations.

Records which demonstrate laboratory performance are filed in categories in a manner similar to project files. Reference documents are not indexed and their usage is not controlled.

The details of maintaining records, documenting laboratory performance are given in current standard operating procedures.

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Table SJ12.2 presents the laboratory operations records to be maintained and the category system of their maintenance. Also shown in this table is the manner in which the files can be correlated to project files, if required.

Many of the laboratory operations records are in daily use, such as the Master Sample Log Book, instrument calibration logs, and control charts. It is not intended that the records be stored daily while they are in use. However, when individual log books, etc., are filled, they are placed in the files. For a book that is in use, it is designated by a file index code and entered into the index.

Index File

The index is provided at the beginning of each category.

A - Master Sample Log Book

The master sample log books chronologically record all samples entering the laboratory, independent of project designation.

B - Instrument Calibration Logs; Data Logs

All calibration performed independent of a specific project are recorded by instrument. A separate file is maintained for each instrument subject to calibration.

C - Instrument Maintenance Logs

Separate maintenance files are kept for each instrument incorporated in the preventive maintenance program. The file includes records of maintenance performed in-house or by outside groups. Service contracts are included in

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the file for the applicable instrument.

D - Computer Software Verification

Copies of the data used to verify performance of software are maintained. For software that is periodically reverified with standard problems, the results of the performance are maintained. A separate file for each software package is indexed.

E - Performance Evaluation Records

Laboratory participation in Performance Evaluation Programs are documented in this category. If performance standards are analyzed as part of the overall Quality Control sample program, the results are cross-referenced to Category G.

F - Certification Program Records

When the laboratory participates in Certification programs, such as the E.P.A. Contact Lab Program, the results shall be maintained in this category. Records include or reference correspondence, analytical data, agency results, and certificates of performance.

G - QC Sample Analysis

This file includes the results of all QC samples inserted into the sample stream. The files are maintained chronologically for each parameter or group of parameters included in the QC program.

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H - Control Charts

Control charts are filed chronologically for each parameter or group of parameters monitored.

I - Purchased Material Certificates

All information which verifies that purchased materials meet the requirements of the laboratory are maintained. Certification is supplied by a vendor or from in-house verification analysis. Separate files are kept for chemicals, gases, water, glassware, etc.

J - QC Coordinator Reports

This file includes the monthly reports prepared by the Quality Control Coordinator. The file is maintained chronologically.

K - Audit Records

The laboratory will maintain records of all audits performed on its facilities and personnel. The file will be maintained chronologically and type of audit performed (Systems, Internal, Governmental, and Customer).

Included in this category; if the audit was project-specific, it is also to be cross-referenced to applicable projects.

L - QC Training Records

Documentation of employee training, both internal and external, will be maintained in this file. Laboratory training plans and matrices will be included. The file will also contain current resumes of employees which

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outline professional qualifications. The information will be maintained alphabetically by employee.

M - Standard Operating Procedures

The mastercopies of laboratory standard operating procedures are kept in this file. Both current and historical SOPs are filed and the first record is an index listing the SOP number, title, and its computer diskette storage. The SOPs will be filed by assigned number.

N - Methods

The general methods used by the laboratory are maintained. The first record in the file is an index listing the method number, title, and its computer-diskette storage. Methods which are specific or alterations for projects are kept with the project records unless the method's use becomes prevalent enough to be kept in this file. The methods are filed by assigned number.

O - Subcontractor Records

Data received from subcontractors from analysis for audit (PE) samples, routine QC samples, blind samples submitted to them, and other information establishing the laboratory's performance levels are maintained in this category by subcontractor and project number.

P - Master Nonconformance Record

Nonconformances and corrective actions which are supplied to the Quality Control Coordinator are filed chronologically. This record is periodically examined and reports issued describing systematic trends which

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in turn require investigation and correction by the responsible Group Leaders, Technical Director, Operations Manager, or General Manager. Group Leaders are encouraged to maintain Group Records to perform such evaluations regularly.

Q - FIFRA Master Projects List (Master Schedule Sheet)

The laboratory maintains a FIFRA Master Projects List in this file as required for its work done under the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) Good Laboratory Practice Standards (40 CFR 160.35). The Quality Control Coordinator has the responsibility for maintaining these files. The files are kept by client, test substance, and study.

SJ12.3 RECORD CONTROL

The General Manager or Operations Manager designates an individual responsible for the records management system. The Quality Control Coordinator is also responsible for certain categories of records. Such responsibilities will be outlined in current standard operating procedures.

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TABLE SJ12-1
PROJECT RECORDS
FILING CATEGORIES

<u>CATEGORY</u>	<u>RECORD DESCRIPTION</u>
-	Project Index
A	Correspondence
B	Chain of Custody
C	Field Records
D	Request for Analysis
E	Raw Data, Instrument Logs, Printouts, Calculations & Series Checklists
E-1	- GC Data
E-2	- GC/MS Data
E-3	- Metals
E-4	- General Chemistry
E-5	- Soils Chemistry
E-6	- Other
F	Analytical Data Summary
G	QC Samples
H	Data Reports
I	Project-Specific Requirements
J	Nonconformances
K	QA Plans
L	Miscellaneous
M	Method Description
N	Work Order Records

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IT ANALYTICAL SERVICES-SAN JOSE
PROJECT CHECKLIST/INDEX

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Client Name: _____ Project #: _____

Work Order #: _____ Analysis: _____

Cat Record Type _____ Included in Project File? _____

A Correspondence & Notes	Yes <input type="checkbox"/>	No <input type="checkbox"/>	List:
B Chain of Custody	Yes <input type="checkbox"/>	No <input type="checkbox"/>	
C Field Records	Yes <input type="checkbox"/>	No <input type="checkbox"/>	
D Request for Analysis	Yes <input type="checkbox"/>	No <input type="checkbox"/>	
E Raw Data, Instr Logs, Printouts, Calcs, & Series Checklists	Yes <input type="checkbox"/>	No <input type="checkbox"/>	See Instr Series <input type="checkbox"/> Referenced on Data Summary Forms (F)
Lab Notebook Copies	Yes <input type="checkbox"/>	No <input type="checkbox"/>	See Instr Series <input type="checkbox"/>
F Analytical Data Summary	Yes <input type="checkbox"/>	No <input type="checkbox"/>	
G QC Samples Results	Yes <input type="checkbox"/>	No <input type="checkbox"/>	See Instr Series <input type="checkbox"/>
H Data Reports	Yes <input type="checkbox"/>	No <input type="checkbox"/>	See Work Order # _____

Special instructions for report:

Proofing copy Yes ☐ No ☐ See Work Order #

I Proj-Specific Requirements	Yes <input type="checkbox"/>	No <input type="checkbox"/>	List:
J Nonconformances	Yes <input type="checkbox"/>	No <input type="checkbox"/>	
K QA Plans	Yes <input type="checkbox"/>	No <input type="checkbox"/>	List:
L Miscellaneous	Yes <input type="checkbox"/>	No <input type="checkbox"/>	List:
M Method Description	Yes <input type="checkbox"/>	No <input type="checkbox"/>	See Instr Series <input type="checkbox"/> Form Lab Notes
N Work Order Records	Yes <input type="checkbox"/>	No <input type="checkbox"/>	AR301144

Signature _____ Date _____

TABLE SJ12-2
 LABORATORY PERFORMANCE RECORDS
 FILING CATEGORIES

CATEGORIES	RECORD DESCRIPTION	CORRELATION TO PROJECT FILES
-	Index File	--
A	Master Sample Log Books	By date of arrival and sample number
B	Instrument Calibration Logs	By date and instrument serial number
C	Instrument Maintenance Logs	By date and instrument serial number
D	Computer Software Verification	By date and software designation
E	Performance Evaluation Records	By program, date and parameter
F	Certification Program Records	By name of program and date
G	QC Sample Analysis	By date and parameter
H	Control Charts	By date and parameter
I	Purchased Material Certificates	By date and parameter
J	QC Reports	By date
K	Audit Records	By date and type
L	Personnel files	By employee and date
M	Standard Operating Procedures	By assigned number
N	Methods	By assigned number
O	Subcontractor Records	By subcontractor and date
P	Master Nonconformance Record	By date
Q	FIFRA Master Project List	By client, test substance, and study

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SJ13.0 NONCONFORMANCES AND CORRECTIVE ACTION

SJ13.3 INTERNAL NONCONFORMANCE CORRECTIVE ACTION PROCEDURE

Any laboratory employee noticing a deficiency suspected of being a nonconformance shall report the deficiency to the responsible supervisor and to the Quality Control Coordinator on a nonconformance memo form. Figure SJ13-1 shows an example of a Nonconformance Memo. The details for reporting nonconformances are given in the current standard operating procedure.

The Quality Control Coordinator maintains copies or a log of nonconformances that includes a description of the problem and corrective action and lists the affected projects and sample numbers.

Once the corrective action has been taken, the Quality Control Coordinator documents that the corrective action has been completed satisfactorily (closed out) by signing the nonconformance memo.

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ITAS - SAN JOSE
NONCONFORMANCE/VARIANCE MEMO

Nº 891173

Memo Date _____ By _____ Analysis affected _____

To be completed by QCC: ☐ Nonconformance ☐ Variance

Filing: Check and Complete One

☐ Project: Client _____ Proj # _____ WO # _____
Project: Client _____ Proj # _____ WO # _____
Project: Client _____ Proj # _____ WO # _____
Project: Client _____ Proj # _____ WO # _____
☐ Instrument Series # _____
☐ Other Equipment _____
☐ Other _____

- Instructions:
1. Fill out completely including description of corrective action.
 2. Put initials and date in appropriate space.
 3. Initial and date all entries.
 4. Copy to QC Coordinator.
 5. Copy to appropriate file as checked above.
 6. Forward original memo immediately.

Title	Initial	Date	Corrective Action Code
Analyst	_____	_____	_____
Team Leader	_____	_____	_____
Group Leader	_____	_____	_____
Ops Manager	_____	_____	_____
QC Coordinator	_____	_____	_____

NONCONFORMANCE:

Describe non-conformance fully; attach documentation if needed.
Initial and date all entries and comments; continue on reverse.

CORRECTIVE ACTION TAKEN:

Check applicable corrective action code; otherwise describe.
Initial and date all entries and comments; continue on reverse.

<input type="checkbox"/> (a) Standard curve drawn	<input type="checkbox"/> (c) Project checklist flagged for
<input type="checkbox"/> (b) Discussed verbally with client	comment in report
Company: _____	<input type="checkbox"/> (d) Client informed with memo
Date: _____ Time: _____	<input type="checkbox"/> (e) 100% check of data
Contact: _____	<input type="checkbox"/> (f) Analyst informed
Discussed all deficiencies? _____	Name _____
Signature: _____	<input type="checkbox"/> (g) Other; describe _____

FIGURE SJ13-1

EXAMPLE - NONCONFORMANCE MEMO

SJ14.0 QUALITY ASSURANCE/QUALITY CONTROL AUDITS

Audits of an analytical laboratory are described as:

- . Performance audits conducted on an on-going basis within the laboratory by the Quality Control Coordinator. These audits are reported to the Operations Manager, Technical Director, General Manager and the ITAS Quality Assurance and Compliance Director. They include: monthly surveillances, and semi-annual internal audits.
- . System audits performed on a scheduled, periodic (semi-annual) basis by the ITAS Quality Assurance and Compliance Director. These audits are external to the laboratory and are reported to the Vice President, Analytical Services and the Corporate Director of Quality Assurance.
- . Data audits performed on representative projects from a laboratory. These audits are conducted on a project-specific basis by the ITAS Quality Assurance and Compliance Director or his representative.

Audits of the laboratory are performed for the following reasons:

- . To determine that contractual and regulatory obligations are fulfilled.
- . To determine that IT procedures and standards are being followed. These audits may include the ITAS QA Manual, this Laboratory-Specific Attachment, project-specific QA Project Plans, Chain of Custody forms, Sample Collection Logs, etc.
- . To establish that Quality Assurance objectives are met, including holding times, reporting turnaround times, use of approved analytical methods, and stated objectives for precision, accuracy, representativeness, completeness, and comparability (PARCCs).

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- . To serve as a management tool to evaluate appropriateness of Quality Assurance policies.
- . To identify potential or actual deficiencies for the purposes of evaluating compliance with requirements and providing the means for correction.
- . To determine that records are prepared/maintained as required.

Audits are not conducted to assign blame.

SJ14.1 PERFORMANCE REVIEWS

To complete internal laboratory reviews, the Quality Control Coordinator provides ongoing monitoring of laboratory operations. The reviews are conducted on behalf of the Operations Manager, Technical Director and General Manager to verify that the laboratory Quality Assurance Program is implemented and functioning on a daily basis. Reviews may be of two kinds: surveillances or semiannual internal audits. Surveillances are inspections of specific laboratory functions as they relate to the QA program, performed on a monthly basis by the Quality Control Coordinator. Surveillances do not require as extensive planning and preparation as do audits, and prior communication with the surveyed group or personnel is not necessary. The Quality Control Coordinator observes the activity of interest while it is in process and/or review objective evidence. The semiannual internal audit covers the same criteria that the semiannual ITAS Quality Assurance Systems Audit does, namely the laboratory's QA Program. The audit is intended to be a spot check and should include the following items :

. Sample maintenance

- Are stated temperatures for sample storage provided?

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- Are samples processed and tested within prescribed holding times?

- Are samples properly logged in?

. Calibration

- Are calibrations performed as required?
- Are they properly documented in instrument log books, or as part of project data if required?
- Do calibration results indicate a trend in instrument performance?

. Preventive maintenance

- Are adequate spare parts available?
- Do specific instruments have repeated maintenance problems?
- Is preventive maintenance performed and properly documented?

. Receipt and storage of standards, chemicals, and gases

- Are all reagents, chemicals, and gases purchased for use in the laboratory of adequate grade for the intended use?
- Are certifications of material compositions provided when required?
- Are materials adequately stored to prevent degradation?
- Are materials kept beyond stated shelf life?
- Are internal standards properly prepared and stored?
- Are internal standards kept beyond stated shelf life?

. Analytical Methods

- Are the methods used appropriate for project requirements?
- Are alternate methods approved for use?

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. Data verification

- Are data processed and validated as prescribed?

. Records management

- Are the records of analyses complete and properly identified?
- Are documents submitted to the record system in a timely manner and are they properly maintained?

Nonconformances observed by the Quality Control Coordinator are documented using a "Nonconformance Memo", reported to the Group Leaders and Operations Manager for corrective action, or the Technical Director, General Manager, or Quality Assurance and Compliance Director, if necessary, for corrective action to be taken. See Section SJ13.0 Nonconformances and Corrective Action.

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SJ15.0 QUALITY REPORTS TO MANAGEMENT

Both the Quality Control Coordinator and the ITAS Quality Assurance and Compliance Director are responsible for preparing reports to management indicating effectiveness of the Quality Assurance Program.

SJ15.1 PERFORMANCE REVIEW REPORTING

The internal laboratory review activities of the Quality Control Coordinator are summarized in a monthly report to the Operations Manager, Technical Director, General Manager and ITAS Quality Assurance and Compliance Director. The report is formatted according to the current policy describing the activities reviewed. To demonstrate review, the Operations Manager, Technical Director and the General Manager shall sign and date the report. The signed copy shall be maintained by the Quality Control Coordinator for a period of two years after the date of the report.

Table SJ15-1 contains example topics for a monthly QA report.

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Table SJ15-1: EXAMPLE TOPICS OF MONTHLY QA REPORTS

1. Audits
 - including auditing party, dates, results, reason for audit, and summary findings
2. Certifications
 - including agency certifying, expiration date, and parameters approved
3. Nonconformances
 - summary of nonconformances and associated corrective action
4. QC Summary Data
 - tables of the QC statistics by analysis and matrix
5. Standard Operating Procedures (SOPs)
 - a listing of SOPs approved or revised
6. Holding Times Violations
 - a listing of samples which exceeded the maximum holding times for the prescribed analysis
7. Training
 - a listing of any QA/QC training received by laboratory personnel and an evaluation of its usefulness
8. Information
 - any pertinent information that might be of use to interested parties
9. QA Project Plans
 - a listing of any Quality Assurance Project Plans (QAPPs) approved by the laboratory including title, client, type of project, and other indicators (methods, parameters, matrices, etc.).

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SJ16.0 TRAINING

SJ16.2.1 Technical Training and Qualifications

An analyst hired to perform sample preparation procedures and/or analytical procedures receives direct instruction from a professional staff member. To become qualified on a particular procedure, the analyst must pass a specified qualifying step under an instructor's supervision. The qualifying is described in the analytical methods or current standard operating procedures. Only upon acceptable completion of the appropriate qualifying step is the analyst able to perform the procedure without assistance or supervision.

SJ16.2.2 Quality Assurance Training and Qualifications

General training in the requirements of the ITAS Quality Assurance Program is required of all laboratory personnel. Formal training sessions are conducted and documented by the Quality Control Coordinator or representative. The training program addresses regulatory requirements as appropriate, basic quality control practices, responsibilities of the technical staff, responsibilities of the QCC, the reporting of nonconformances and the performance of audits. In addition, each laboratory analyst should become familiar with the laboratory quality assurance program by reading quality control procedures and sections of this laboratory-specific attachment and the ITAS QA Manual appropriate to his/her position.

SJ16.4 QUALIFICATION AND TRAINING RECORDS

Each laboratory employee has a Personnel Qualifications Record. The record is documented with technical and quality assurance procedures for which the employee is qualified, the dates of qualification and renewal, and the

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approval signature. Details for the records are given in current standard operating procedures.

The Quality Control Coordinator is responsible for maintaining the personnel qualifications and training records as quality documents in the Quality/Operations record system Category N.

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